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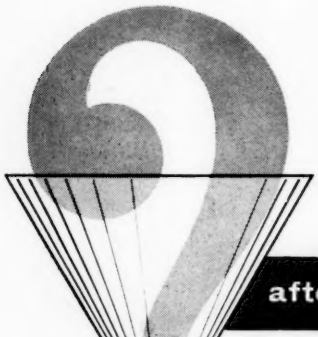

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

2057. **Chemical analysis in the future.** C.-J. van Nieuwenburg. *Chim. Anal.*, 1956, **38** (12), 423–427.—A forecast. W. J. WRIGHT

2058. **Application of cryoscopy in analytical chemistry.** J. Dykyj (Výskumný ústav acetylenovej. chémie, Nováky). *Chem. Prûmysl.*, 1956, **6**, 52–54.—The use of cryoscopic methods for technical analysis of binary and ternary mixtures is discussed and illustrated with reference to vinyl acetate - acetic acid and diethylene glycol - triethylene glycol - water mixtures. S.C.I. ABSTR.

2059. **Removal of phosphorus from hydrogen peroxide by kaolinite.** S. C. Chang and M. L. Jackson (Dept. of Soils, Univ. of Wisconsin, Madison, U.S.A.). *Science*, 1956, **124**, 1209.—The common contamination of H_2O_2 with phosphoric acid necessitates its purification before use as an oxidising agent in determining organic P. The following procedure reduces the content of P to between 0·1 and 0·2 p.p.m. Shake 100 ml of H_2O_2 (30%) by hand for 5 min. with 10 g of kaolinite, allow to stand for 1 min. and decant through a Buchner funnel under gentle suction. Treat the filtrate with kaolinite and repeat the treatment 3 or 4 times; clarify the final filtrate by centrifuging and add 0·5 ml of conc. HCl to each 100 ml of purified H_2O_2 for stabilisation. H. F. W. KIRKPATRICK

2060. **The basis of the selectivity of dimethylglyoxime as a reagent in gravimetric analysis.** A. G. Sharpe and D. B. Wakefield (Univ. Chem. Lab., Cambridge, England). *J. Chem. Soc.*, 1957, 281–285.—The selectivity of dimethylglyoxime as a precipitant for Ni, Pd and Pt is based on the unique planar structure of the molecules of these complexes, which lie parallel to each other and are bonded together by weak metal-to-metal links. The only other two metals likely to form planar 4-co-ordinate complexes are Cu and Co, both of which form soluble complexes. This solubility is due, in the case of Co, to what is probably an octahedrally co-ordinated Co^{2+}, which precludes metal-to-metal bonding; and to two firmly bonded molecules of water, which means that unlike Ni the phase in equilibrium with a saturated soln. of the cobalt complex is a stable dihydrate. The complexes of Cu and Ni have very similar structures and mol. dimensions, but the arrangement of the mol. in the copper lattice precludes metal-to-metal bonding. Evidence for the presence of strong but not symmetrical hydrogen bonds in the copper complex presents a paradox, since they would be expected to have a desolubilising effect. It is concluded that this factor plays only a subsidiary part in determining solubility. G. S. ROBERTS

2061. **o-Hydroxyacetophenone as an analytical reagent. I.** Sailendra Nath Poddar (Indian Ass. for the Cultivation of Science, Jadavpur, Calcutta). *Z. anal. Chem.*, 1957, **154** (4), 254–259 (in English).—The reactions of *o*-hydroxyacetophenone oxime with metal ions, the preparation of the reagent, and the preparation, properties and structure of the complexes are presented and compared with those of salicylaldoxime, of which it is the methyl derivative. It is shown to be useful for the gravimetric estimation of Cu, Ni, Pd and V, and for the colorimetric estimation of Fe, U and V. G. BURGER

2062. **Resazurin and some of its derivatives as analytical reagents.** E. Ružička (High Pedagog. School, Olomouc, Czechoslovakia). *Chem. Listy*, 1957, **51** (1), 173–175.—Resazurin, ethoxyresazurin (**I**), acetoxyresazurin (**II**) and tetrabromoresazurin (**III**) were studied as analytical reagents. The red or reddish-brown soln. of these dyes are reduced by Sn^{2+} in HCl to greenish-blue or colourless soln., and Ti^{3+}, Cr^{2+}, S^{2-} and $S_2O_3^{2-}$ in HCl or in 4 *N* H_2SO_4 react similarly, as do I^- and SCN^- in conc. H_2SO_4. These reactions can be used for the detection of Ti^{3+}, Cr^{2+}, S^{2-}, I^- and SCN^- with **I**, and Sn^{2+} and $S_2O_3^{2-}$ with **II**. When detecting these compounds in the presence of Fe^{2+}, the use of **III** is preferable. Procedures for carrying out the reactions in the test-tube or on paper are described, and their sensitivity is given. J. ZÝKA

2063. **Disodium 1:8-dihydroxynaphthalene-3:6-disulphonate (chromotropic salt) as a reagent in inorganic analysis. Colour reactions with metal ions.** K. C. Mathur and A. K. Dey (Univ. of Allahabad, India). *Z. anal. Chem.*, 1957, **154** (5), 347–351 (in English).—Chromotropic salt (B.D.H. indicator) is widely used as a reagent for Ti in acid medium. The colour reaction of the reagent (0·1% aq. soln.) with 40 metal ions in neutral, alkaline and acidic media has been investigated. It was found to be particularly sensitive in producing coloured complexes with U^{II}, Fe^{III}, Hg^{I}, Sn^{IV}, Au^{III} and Mo^{VI}, and sensitive with Pt^{IV}, Te^{VI}, Al^{III} and Cr^{III}, as well as with V and Ti. Beer's law was obeyed with U and Fe and is being investigated with other metal ions. D. F. PHILLIPS

2064. **Use of some thiolactic anilides in inorganic analysis.** R. N. Misra and S. S. Guha Sircar (Mayurbhanj Chem. Lab., Ravenshaw College, Cuttack). *J. Indian Chem. Soc.*, 1956, **33** (7), 523–526.—Six thiolactic anilides (thiolactic anilide, *o*-toluidide, *p*-toluidide, *o*-chloroanilide, *m*-chloroanilide and *p*-chloroanilide) have been prepared and their usefulness in analysis studied. In most cases, Ag, Pb, Hg^{I}, Hg^{II}, Cu^{II}, Cd, Bi, Sb, Fe^{III}, Ni, Co, Zn and molybdate form complexes. The sensitiveness for Ni and Co has been determined. The nickel complex of thiolactic *o*-toluidide contains two mol. of reagent per atom of Ni. I. JONES

2065. **Qualitative inorganic analysis. I. (Introduction.)** R. Belcher (Dept. of Chem., Univ., Birmingham, England). *Mikrochim. Acta*, 1956, (12), 1842–1846 (in English).—The reasons for setting up the Midlands Qualitative Inorganic Analysis Committee in 1954 are briefly reviewed. The committee consists of representatives from advanced teaching institutions and has as its object the compilation of a reliable scheme for semi-micro analysis and the examination of conflicting statements made in text books. A series of notes and papers is to be published in *Mikrochim. Acta* before the final recommended scheme is made available.

II. A semi-systematic scheme for the detection of anions. R. Belcher and H. Weisz. *Ibid.*, 1956, (12), 1847–1855 (in English).—Of the 36 (or 39) anions for which the scheme is designed, 24 are detected directly by separating them into a number of groups and applying identification tests without further separation. The other anions are identified by special tests. The limitations of systematic schemes are discussed. P. S. STROSS

2066. **Improvements in methods of rapid qualitative analysis. II.** A. Schleicher (Rheinisch-Westfälischen Tech. Hochschule, Aachen). *Mikrochim. Acta*, 1956, (12), 1747–1750 (in German).—The author discusses comments and criticisms of his earlier paper [*cf. Mikrochim. Acta*, 1956, (4–6), 663]. The future of qualitative analysis does not lie in the perfection of schemes for the systematic identification of all the elements, but in a great increase in the sensitivity of existing methods, enabling trace analyses of all the elements to be made. The requirements by which these methods will be evaluated are briefly discussed. P. S. STROSS

2067. **Inorganic spot tests. I. Identification of thallium.** F. Feigl and D. Goldstein (Mineral Prod. Lab., Min. Agric., Rio de Janeiro). *An. Ass. Brasil Quim.*, 1953, [1956], **12** (1), 13–15.—A spot test for Tl depends on oxidation of Tl^+ to Tl^{3+} with bromine water, removal of excess of Br with sulphosalicylic acid, and the addition of $Cd(IO_3)_2$ soln. containing starch; a blue colour indicates Tl. If NO_3^- are present a little urea is added after the sulphosalicylic acid. The limit of identification is 0·2 μg of Tl. A second test depends on the formation of TlO(OH) by the addition of ferricyanide to an alkaline soln. A brown turbidity appears if Tl (>10 μg) is present. Smaller amounts (>0·5 μg) may be detected by adding benzidine, when a blue colour appears.

II. Identification of palladium. F. Feigl, D. Goldstein and W. A. Mannheimer. *Ibid.*, 1953, [1956], **12** (1), 17–18.—A drop of soln. of HgI_2 in dioxan is treated with a drop of a slightly acid soln. containing Pd. According to the concn. of Pd, either a black or grey ppt. is formed.

III. Identification of platinum. F. Feigl and V. Gentil. *Ibid.*, 1953, [1956], **12** (1), 19–20.—Platinum salts react with alkali iodide in neutral or acid soln. to give a red - brown colour of the complex $(PtI_6)^{2-}$, which is destroyed by alkali sulphite. Interference by salts of Pd or Au can be eliminated by treatment with aq. NH_3 and oxalic acid.

IV. Use of sulphosalicylic acid to remove free bromine. F. Feigl, D. Goldstein and W. A. Mannheimer. *Ibid.*, 1953, [1956], **12** (1), 21–23.—Sulphosalicylic acid is a better reagent for the removal of free Br than is phenol or formic acid. H. PRITCHARD

2068. **Complete analysis of anions by paper chromatography. IV.** Zenzo Tamura and Motoichi Miyazaki (Pharm. Inst., Med. Fac., Tokyo Univ., Hongo, Tokyo). *Japan Analyst*, 1956, **5** (10), 566–570.—The separation of 20 anions (sodium salts) was studied with three developers. Halogens, SCN^- (R_F 0·05), ClO_3^- (0·65), BrO_3^- (0·38) and NO_3^- (0·45) were developed with acetone containing water (10 + 1), and detected by spraying with $AgNO_3$ soln., followed by exposure to light (for halogens), $FeCl_3$ (for SCN^-) and acid KI soln. (for the remaining anions). Fluoride, NO_2^-, $S_2O_3^{2-}$, SO_4^{2-}, CrO_4^{2-}, IO_3^-, AsO_3^{3-}, $Fe(CN)_6^{4-}$, $Fe(CN)_6^{3-}$ and BO_2^- were developed with a mixture of *n*-butanol, methanol and water (1 + 3 + 1) and revealed with the ordinary colour reagents (Ba^{2+} and Na rhodizonate for SO_4^{2-} and $S_2O_3^{2-}$). Ferricyanide is partly reduced to $Fe(CN)_6^{4-}$ during development. Nitrite volatilises under the given conditions. Oxalate, AsO_4^{3-} and PO_4^{3-} were developed with *n*-butanol satd. with 2 *N* HNO_3, and revealed with $Fe(SCN)_3$ (for oxalate), $(NH_4)_2MoO_4$ and benzidine acetate. K. SAITO

2069. **Influence of the volume of the precipitate in precipitation reactions in quantitative analysis.** B. Buděšínský (Res. Inst., Pharm. and Biochem., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (1), 166–168.—A pptn. reaction, when carried out in a definite vol. of the soln. of which an aliquot is measured for further procedure, can be influenced by the vol. of the ppt. itself. A detailed study of this phenomenon is presented and mathematically evaluated, and the validity of derived mathematical relations is confirmed experimentally. J. ZÝKA

2070. **Strontium thiosulphate monohydrate: a useful standard in iodimetry.** W. L. W. Ludekens and N. R. de Silva (Dept. of Chem., Univ. of Ceylon, Colombo). *Research*, 1956, **9** (8), s31–s32.—The efflorescence of $Na_2S_2O_3.5H_2O$ makes it unsuitable as a standard in iodimetry. Strontium thiosulphate monohydrate does not effloresce and can be obtained in 99·90% purity by adding the theoretical amount of $Na_2S_2O_3$ to a saturated soln. of $SrCl_2$ at 60°; slow evaporation at the same temp. in a desiccator containing conc. H_2SO_4 yields well-formed crystals, which are washed with a little water and dried in a desiccator containing $CaCl_2$. Titration with iodine soln., standardised against As_2O_3, confirmed the formation of the monohydrate, also the normality of an iodine soln. determined against this salt agreed closely with the figure obtained by using As_2O_3. The salt is stable in humid atmospheres, and has a high equiv. wt. and an appreciable solubility. P. HAAS

2071. **2:5-Di-(2-hydroxyethylamino)terephthalic acid as indicator for argentimetric titrations.** E. Uhlig (Inst. anorg. Chem., Karl Marx Univ., Leipzig). *Z. anorg. Chem.*, 1956, **288**, 24–27.—During the course of investigations of benzenedicarboxylic acids it was found that 2:5-di-(2-hydroxyethylamino)terephthalic acid (**I**) could be readily oxidised by the salts of Ag, Hg and Fe^{III} to an intense red water-soluble product. The reaction was very sensitive, and was applicable to the determination of chlorides and bromides. *Procedure for bromides*—To a neutral or slightly acid solution of the bromide was added **I** (5 to 10 mg). The mixture was titrated, with stirring, with 0·1 *N* $AgNO_3$. At the end-point a distinct red coloration appeared. Results agreed to within 0·2% with those obtained by a Volhard titration.

Chlorides—The procedure was as for bromides, except that $NiSO_4.7H_2O$ (500 mg) was added at the start. At the end-point the solution turned from pale green to red - violet. The nickel salt was added to prevent adsorption of the oxidation product on the AgCl ppt. The accuracy here was also within 0·2% of results obtained by other methods. Iodides cannot be accurately determined owing to co-pptn. with the AgI of both iodide and silver ions. Copper forms a ppt. with the indicator. The chlorides and bromides of Zn, Ba, Ni and Al may be determined without further precautions. L. S. ADLER

2072. **Xylenol orange: new indicator for the EDTA titration.** J. Körbl and R. Přibl (Inst. Chem., Czechoslovak Acad. Sci., Prague, Czechoslovakia). *Chemist Analyst*, 1956, **45** (4), 102–103.—Xylenol orange, [6-di(carboxymethyl)aminomethyl)-*o*-cresol]sulphonephthalein (**I**), retains the acid - base properties of *o*-cresolphthalein and acts as a metal indicator even in acid soln., giving a distinct colour change in direct EDTA titration. Examples of its use are given. The results of the titrations are very accurate. In soln. as acid as *M* HNO_3, and at 90°, Zr may be titrated with EDTA soln. By appropriate pH adjustments, certain pairs of metals, *e.g.*, Bi and Zn, Bi and Pb, or Bi and Cd may be titrated successively in a single sample soln. The procedures described are also suitable for use on a micro-scale. O. M. WHITTON

2073. **The EDTA titration: nature and methods of end-point detection. I.** A. J. Barnard, jun., W. C. Broad and H. Flaschka (J. T. Baker Chem. Co., Phillipsburg, N.J., U.S.A.). *Chemist Analyst*, 1956, **45** (4), 86–93, 111–112.—This review deals with the evolution and theory of the method, the reagent and its physical properties and nomenclature, the nature of the EDTA titration, including effect of pH, stability constants, types of titrations, selectivity through masking, etc., visual end-point detection by indicators, and the prep., physical properties and other laboratory uses of the metal indicators. (127 references.) O. M. WHITTON

2074. **Micro-titration with ethylenediaminetetra-acetic acid. XVI. Metal determination in the ultra-micro range.** H. Flaschka and F. Sadek (Nat. Res. Centre, Dokki-Cairo, Egypt). *Mikrochim. Acta*, 1957, (1), 1–18 (in German).—The titration of a few microlitres of 0·001 *M* metal salt soln. in approx. 1 ml of titration vol. is described. Details and necessary precautions are given which are applicable for any titration in the ultra-micro range and enable an end-point reading better than ± 1 μl to be attained. Special emphasis is laid on the avoidance of error due to glassware and reagent contamination. Eriochrome black T is used as complexiometric indicator for the titration of Mg, Ca, Mn, Zn and Cd; PAN [1-(2-pyridylazo)-2-naphthol] for Cu, Ni, Pb, Fe and Zn; and catechol violet for Bi and Th. D. F. PHILLIPS

2075. **Use of organic sulphur derivatives in volumetric analysis. III. Titrations with mercaptobenzothiazole.** J. Čihalík and E. Kudrnovská-Pavlíková (Inst. Anal. Chem., Charles' Univ., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (1), 76–81.—Methods were developed for the amperometric determination of Ag^+, Hg^+, Hg^{2+} and Bi^{3+} with a soln. of mercaptobenzothiazole (**I**), and also for the determination of **I** with a soln. of $AgNO_3$. The method for Ag is applicable to micro-determinations. For all titrations the dropping-mercury electrode was used as indicator electrode; for the titration of Ag^+, Hg^+ and Hg^{2+} the short circuit system was used; and for the titration of Bi^{3+} an e.m.f. of − 0·35 V was used; 0·5 *N* KNO_3 was a suitable supporting electrolyte. The titrations must be carried out under the following conditions—Ag (2 to 10 mg): pH 3 to 7, vol. 20 to 30 ml; Hg^+ (5 to 10 mg): pH 5·6, vol. 15 to 20 ml; Hg^{2+} (6 to 10 mg): pH 4·5 to 5, vol. 30 ml; Bi^{3+} (1·5 to 10 mg): pH 4 to 5, vol. 30 ml. In a vol. of 10 ml, as little as 200 μg of Ag can be determined. The average errors are—Ag ± 0·48%, Hg^+ ± 0·39%, Hg^{2+} ± 0·44%, Bi^{3+} ± 0·47%. The titration of **I** can be carried out in aq. NH_3 soln. (e.m.f. −0·4 V) or in neutral ethanolic soln. (short circuit system) with an average error of ± 0·48% for 5 to 60 mg of the sample. J. ZÝKA

2076. **New methods of gas analysis. III. Physical and chemical properties of alcohol - water systems.** E. Schulek, E. Pungor and J. Trompler (Eötvös Univ., Budapest, Hungary). *Mikrochim. Acta*, 1957, (1), 85–95 (in German).—Measurements are reported of tension, viscosity and other physical constants of various alcohol - water systems. From these results and from literature data it is concluded that the deviations from ideal conditions that were found are functions of molecular arrangement, and that molecular arrangement varies with component concn. D. F. PHILLIPS

2077. **Interpretation of areas used for quantitative analysis in gas - liquid partition chromatography.** L. C. Browning and J. O. Watts (U.S. Naval Powder Factory, Indian Head, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 24–27.—Gas - liquid partition chromatography was applied to the quant. analysis of water - ethanol - diethyl ether, CCl_4 - acetone, ethanol - $CHCl_3$ and CCl_4 - $CHCl_3$ mixtures. In soln. where the difference in thermal conductivities of the components is small, areas under the elution curves obtained may be used to determine wt.-% directly. In cases where the thermal conductivities differ considerably, an empirical calculation can be made by dividing the individual areas by the respective thermal conductivity and calculating the wt.-% from these values. G. P. COOK

2078. **Focusing ion exchange. I. Principle and simple theory.** E. Schumacher (Zurich Univ., Switzerland). *Helv. Chim. Acta*, 1957, **40** (1), 221–228.—Homogeneous ion exchange between cations and complex anions produces separation effects in an electric field. If this field is maintained across a stationary gradient of the complex-forming anions it is shown that participating cations will be focused into a system of narrow zones on a column. There will be as many zones as there are pure cationic species. The theory of this new method of separation, which it is claimed is not realised on resin columns, is developed semi-quantitatively, and the parameters of the method are discussed. In order to avoid convection effects due to the heat developed by the electric current it is proposed to use columns, *e.g.*, filter-paper strips, which can be cooled. The electrical field strength determines the migration velocity of the ions and hence the rapidity of focusing. This field strength should therefore be as high as possible, *e.g.*, up to 100 V per cm. Several possible applications are proposed for investigation.

II. Rapid qualitative cation analysis. E. Schumacher and H. J. Streiff. *Ibid.*, 1957, **40** (1), 228–233.—This technique of focusing ion exchange

is used for the qualitative separation of cations from mixtures containing from 1 to 10 μg of each ion. Separation is accomplished in 5 min. and the components are concentrated into so small a volume that they can subsequently be detected by conventional reagents. The apparatus is simple and cheap. A filter-paper strip (Whatman No. 1), 2 cm wide, is used as a separating column. One drop (0·05 ml) of ion mixture in the form of nitrate or chloride is applied to the previously moistened central zone of the paper over a length of 2 to 3 cm. This zone is immersed in a CCl_4 cooling bath. One end of the strip dips into an anode soln. and the other into a cathode soln. Platinum or carbon electrodes are used for both electrode soln. A voltage of approx. 200 V with a current of about 10 mA is applied to the electrodes immediately the electrode zones have connected with the test zone by capillary action. The gradient of complexed ions is established in about 2 min. and focusing of the separated zones is complete within a further 3 min. The paper strip is removed, dried and treated with the usual colour-forming reagents. Results are reported for the separation and identification of groups 2, 3 and 4, with a pH gradient, and EDTA (disodium salt), nitrilotriacetate, acetate, chloride and tartrate as complexing agents.

III. Rapid separation of radioactive tracers. E. Schumacher and H. J. Streiff. *Ibid.*, 1957, **40** (1), 234–236.—This separation was first studied with the aid of approx. 1 μC of ^{89}Sr (containing a slight unknown amount of ^{90}Sr in radioactive equilibrium with its daughter ^{90}Y) to which was added 20 μg of inactive Sr. This was followed by the successful quantitative separation of a carrier-free sample (≃1 μC) of ^{90}Sr from ^{90}Y. The results were evaluated by both autoradiography and counting, which gave the same values. The radiochemical purity of the ^{90}Sr in its separated zone was shown to be <99%, and the separation was accomplished in a few minutes. D. F. PHILLIPS

2079. **Cation-exchange properties of phenol-formaldehyde resins as affected by the type of acid groups.** E. B. Trostyanskaya, I. P. Losev and A. S. Tevlina (D.I. Mendeleev Chem.-Tech. Inst.). *Zhur. Anal. Khim.*, 1956, **11** (5), 578–584.—A review of work published by the authors and others. (20 references.) G. S. SMITH

2080. **Electro-migration on filter-paper in the separation of ions.** H. Gopal Mukerjee (Scottish Church College, Calcutta, India). *Z. anal. Chem.*, 1957, **154** (5), 344–347 (in English).—A study was made of the migration sequences of Zn and Mn, their separation from each other and from other ions such as Ni, Co, Ag, Cu, Pb, Hg and Cd in different electrolytes. Durrum's simple hanging-strip type of apparatus was used, with a Whatman No. 2 strip (1 cm × 42 cm) touching the electrolyte in each of two glass vessels. These vessels were covered with a plastics sheet having slits for the strip and small holes for the platinum electrodes. A current (d.c., 150 V) was passed for 5 hr. with binary and ternary mixtures and for 10 hr. with quaternary and sexternary mixtures. After drying and developing the paper strip with specific reagents for location of the zone of the separated ions, it was shown that ions of Zn and Mn from different mixtures had been effectively separated. Six cations from a mixture of Pb, Co, Zn, Cd, Cu and Hg could not be completely separated, but were easily identified. D. F. PHILLIPS

2081. **The present status of high-resolution NMR spectroscopy in analysis.** E. B. Baker (Dow Chemical Co., Midland, Michigan, U.S.A.). *Appl. Spectroscopy*, 1956, **10** (4), 198–202.—This review deals with the principles and applications of nuclear magnetic resonance spectroscopy to compound and structure identification, as well as to elementary analysis and the quant. analysis of mixtures. (33 references.) K. A. PROCTOR

2082. **Chronopotentiometric analysis in fused lithium chloride - potassium chloride.** H. A. Laitinen and W. S. Ferguson (Univ. of Illinois, Urbana, U.S.A.). *Anal. Chem.*, 1957, **29** (1), 4–9.—The determination of salts dissolved in high-temp. melts can be made by chronopotentiometric analysis by using platinum micro-electrodes without physically constrained diffusion fields and with transition times (t) of <5 sec. (*cf. Anal. Abstr.*, 1955, **2**, 2329; 1956, **3**, 13). Measurements on $BiCl_3$, $CdCl_2$, AgCl and CuCl dissolved in a LiCl - KCl eutectic at 450° confirm the validity of the theoretical equations relating c.d., concn. and transition time. The diffusion coeff. of Bi^{3+}, Cd^{2+}, Ag^+ and Cu^+ are, respectively, 0·6, 1·7, 2·6 and 3·5 × 10^{-5} sq. cm per sec. The method, for which the accuracy is within ±2·6%, can be applied also to mixtures of $BiCl_3$ - AgCl and $BiCl_3$ - CuCl. The c.d. ($i^{0.5}$) *vs.* concn. curves for the above-named salts (2 to 70 mM) are shown and discussed. W. J. BAKER

2083. **Fluorimetry. II. Use of short-wave ultra-violet rays in analysis.** J. Eisenbrand and D. Pfeil (Chem. Untersuch. für das Saarland, Saarbrücken, Germany). *Z. anal. Chem.*, 1957, **154** (3),167–170.—Short-wave u.v. rays can be used for the chromatographic detection of compounds that possess high light absorption, even if not fluorescent. Some inorganic salts, *e.g.*, $NaNO_3$, NaI, $NaNO_2$ and $Na_2S_2O_3$ in concn. of 10, 1, 1 and 1%, respectively, uniformly gave dark-blue spots with, and greyish-black spots without, a filter at 253·7 mμ; none of these, except $NaNO_2$, gave spots at 366 mμ. Salts not capable of light absorption at 253·7 mμ, *e.g.*, Na_2SO_4, NaCl, NaBr and Na_2HPO_4, show no dark spots, but various org. compounds in soln. of 1 or 10 mg-% that show no or only a weak fluorescence give dark spots. Some fluorescent compounds, *e.g.*, salicylic acid, can be identified by this method, the spots being bright instead of dark. The rise in the intensity of their fluorescence corresponds to the increase in light absorption at 366 mμ. A graph is given for the u.v. absorption spectra of salicylic acid and its sodium salts. (See also *Anal. Abstr.*, 1957, **4**, 271.) W. J. WRIGHT

2084. **Reproducibility of radioactive-sample preparation techniques.** G. K. Schweitzer and J. S. Eldridge (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chim. Acta*, 1957, **16** (2), 189–193 (in English).—Factors affecting the reproducibility of radioactive-sample prep. by the direct evaporation of a soln. of a radionuclide on a shallow container were investigated for ^{35}S, ^{32}P and ^{137}Cs - Ba. The effect of high concn. of total solids on the reproducibility of measurements made with the prepared sample is much greater for low-energy β-emitters than for higher-energy ones. The use of a single-channel scintillation spectrometer for counting γ-emission gives better reproducibility at high concn. of solids than does a β-detector for counting β-particles. Copper or aluminium dishes give the max. reproducibility with the β-emitters, but copper dishes or steel cups are best for the γ-emitter. The pH of the

soln., the use of supplementary porous-paper discs or of a wetting agent are without appreciable effect on the reproducibility of the samples prepared.
W. J. BAKER

2085. **The mass spectrometer in chemical analysis.** J. D. Waldron (Metropolitan-Vickers Electrical Co. Ltd., Manchester). *Research*, 1956, **9** (8), 306–316.—The methods and applications of the mass spectrometer are reviewed with special reference to its use for the qual. identification of org. compounds, the quant. analysis of both inorg. and org. mixtures, high-mol.-wt. spectroscopy and recent developments in techniques for the analysis of impurities in solids. Factors affecting the accuracy of analysis are considered, and mass spectrometry is compared with other analytical techniques. P. HAAS

See also Abstract 2124.

2.—INORGANIC ANALYSIS

2086. **Investigation of chemical composition in a micro volume of an alloy.** I. B. Borovskiĭ and N. P. Il'in (A. A. Baikov Inst. of Metallurgy, Acad. Sci., USSR, Moscow). *Dokl. Akad. Nauk, SSSR*, 1956, **106** (4), 655–657.—Equipment permitting of the surface analysis of alloys in volumes of 10 μ^3 is described. A narrow beam of electrons is focused on an area 2 to 4 μ in diam. on a polished etched anode in a vacuum tube, with a built-in microscope permitting visual scanning of the surface. The anode is moved mechanically at rates of 10 to 120 μ per min., allowing the scanning of the surface, micro discontinuities in which are followed from variations in frequency of characteristic X-radiation emitted by elements contained in the phases of the alloy. The method is particularly suitable for the analysis of inclusions. R. TRUSCOE

2087. **α-Methoxyphenylacetic acid as reagent for detection of sodium ion.** W. Reeve and I. Christoffel (Univ., College Park, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 102–105.—The detection of Na^+ in the presence of Cl^-, NO_3^-, PO_4^{3-} and SO_4^{2-} and of large concn. of salts of ammonium, Li, K and Mg can be effected with an aq. ethanolic soln. of α-methoxyphenylacetic acid (**I**) half-neutralised with tetramethylammonium hydroxide. The sodium acid salt of **I** is almost insol. in H_2O and ethanol at 0°, and the lower limit of detection is ≃0·3 mg per ml. Calcium, Ba and Sr interfere, as does Cs, which behaves like Na. Two equally sensitive procedures are given: (*i*) the test soln. is kept at 15° for ≃1 hr., (*ii*) when room temp. differ much from 15° the soln. is kept at 0° for 1 to 2 hr., raised to 20° and stirred (5 min.) to dissolve any other pptd. salts. Interfering ions, sensitivity and confirmation of the identity of the ppt. are discussed.
W. J. BAKER

2088. **Gasometric method for the determination of sodium in metallic sodium.** Shinzo Okada, Nobuatsu Watanabe and Yuichi Omote (Dept. Ind. Chem., Faculty of Engng, Kyoto Univ., Sakyo-ku, Kyoto). *Japan Analyst*, 1956, **5** (10), 562–566.—The determination of the purity of metallic Na was studied by the use of a gasometric method, involving a reaction between methanol and Na. The sample (≃20 mg) in a stoppered tube is caused to react with methanol (≃20 ml) to yield H (≃10 ml), equiv. in amount to the metallic Na. Conc. H_2SO_4 (>85%) is the best confining liquid. Correction of the vol. measured at a given temp. and pressure can be achieved by the use of an empirical table.
K. SAITO

2089. **Volumetric determination of sodium content in industrial salt.** Fumikazu Kawamura and Hiroshi Namiki (Faculty of Engng, Yokohama Univ., Minami-ku, Yokohama). *Japan Analyst*, 1956, **5** (9), 505–508.—The sample soln. (≃0·5 g in 100 ml) is treated with a known amount of 2% $Ba(OH)_2$ to precipitate $BaSO_4$, which is filtered off, and the filtrate is boiled with a mixture of Ag_2O (2·0 g) and Ag_2CO_3 (1·5 g). Chloride is quantitatively converted into the silver salt, and Ca, Mg and the excess of Ba are pptd. as carbonate or hydroxide and filtered off. The filtrate, containing NaOH (and KOH), is titrated with 0·2 *N* HNO_3, with bromophenol blue as indicator; the amount of added $Ba(OH)_2$ is deducted. When the amount of Ag_2CO_3 is much more than that of Ag_2O, the pptn. of Mg is not quantitative, whilst that of Cl^- is incomplete in a deficiency of CO_3^{2-}. The error is <0·3%. Potassium must be determined by some other method and its amount deducted from the titration value.
K. SAITO

2090. **Determination of sodium in aluminium-copper alloys with the flame photometer.** H. F. Hourigan and J. W. Robinson (P.O. Engng Dept., Birmingham, England). *Anal. Chim. Acta*, 1957, **16** (2), 161–164 (in English).—The rapid routine method described previously (*Anal. Abstr.*, 1956, **3**, 34) has been extended to aluminium alloys containing ≯ ≃ 5% of Cu and up to 100 p.p.m. of Na. *Procedure*—Dissolve the alloy (1 g) in 15 ml of conc. HCl and dilute with water to 100 ml. Centrifuge to precipitate any silicon and determine Na in the supernatant liquid with a flame photometer, using a Na filter and a calibration curve prepared with soln. containing approx. equiv. amounts of Al and Cu. Correct for a blank on the HCl and water used. The accuracy is satisfactory.
W. J. BAKER

2091. **Spectrographic determination of sodium and potassium in silicate minerals.** Ê. E. Vaĭnshteĭn and V. V. Korolev. *Zhur. Anal. Khim.*, 1956, **11** (5), 627–633.—With minerals containing <6% of Na and K the powdered sample is mixed with Li_2CO_3, CuO and C and excited in an a.c. arc. The intensities of the lines K 7664·9 A and Na 8194·8 A are compared with that of Li 8126·5 A. With minerals containing higher contents of Na and K, similar mixtures are used, but line widths are measured. G. S. SMITH

2092. **Rapid method for determining potassium and sodium in coal ash and related materials.** P. J. Jackson and A. C. Smith (Central Electric Authority, Res. Lab., Randalls Farm Lane, Leatherhead, Surrey). *J. Appl. Chem.*, 1956, **6** (12), 547–559.—The flame-photometric method, which is quicker and demands less skilled manipulation than classical methods, has the disadvantage that the presence of calcium salts results in inaccurate readings. The effect of adding Al, Ca, Fe, silicate and borate was studied with the EEL photometer. Only Ca caused significant interference, and this was inhibited (in the determination of Na) by keeping it in the form of sulphate with an excess of H_2SO_4, and by adding Al so that the ratio of total Al to Ca was at least 3. F. J. M.

2093. **New gravimetric method for the rapid estimation of copper.** D. Pirtea, E. Buzatu and S. Zelig. *Stud. Cercet. Chim., Bucharest*, 1955, **3** (3–4), 233–236.—Copper is pptd. quant. as $[CuPy_4](ClO_4)_2$ by the addition of pyridine and solid $NaClO_4$ at room temp. After filtration, the ppt. is finally washed with ether and is dried in a vacuum desiccator. The analysis can be performed in <45 min.
J. H. WATON

2094. **New rapid and accurate method for the gravimetric estimation of copper.** G. Spacu and E. Antonescu (Academ. R.P.R., Bucharest). *Stud. Cercet. Chim.*, 1955, **3** (3–4), 161–165.—Copper is quant. pptd. by the addition of aniline and NH_4SCN soln. as the complex $[Cu(aniline)_2(SCN)_2]$. After filtration, the ppt. is washed with ethanol and with ether, and is dried in a vacuum desiccator. The relative error of the determination is $< \pm 0{\cdot}07\%$. The method allows Cu to be separated from Fe and Al if Fe and Al are masked with Na sulphosalicylate.
J. H. WATON

2095. **Inorganic analysis in organic solvents. II. 8-Hydroxyquinaldine as a reagent for the determination of copper.** A. J. Blair and D. A. Pantony (Roy. School Mines, London, England). *Anal. Chim. Acta*, 1957, **16** (2), 121–128 (in English).—The procedure given applies only to soln. of alloys, ores, etc., containing from 0·01 to 1 mg of Cu. The Cu (with other metals) is removed completely from the aq. soln. (pH 5 to 8) of its 8-hydroxyquinaldate by three extractions into $CHCl_3$. The extract (concentrated to 5 to 10 ml) is diluted with an equal vol. of benzene and passed through a 12 to 15-cm column of activated Al_2O_3 whereon all the quinaldates (except Cr) are adsorbed. The Cu is then quant. eluted from the column with 0·5% 8-hydroxyquinoline in benzene - $CHCl_3$ (1 + 1), and the extinction of the coloured eluate (50 ml) is measured at 400 mμ in a 1-cm cell with respect to the solvent (a 4-cm cell is used when the concn. of Cu is $<$ 0·15 mg). Concn. of $>0{\cdot}5$ mg of Fe, $>0{\cdot}2$ mg of Mo or $>0{\cdot}01$ mg of V cause interference, but this can be obviated or minimised by ether extraction from an HCl soln. of the original sample. If $>$ 0·05 mg of Cr is present, the $CHCl_3$ extract should be evaporated to dryness, and the residue heated at 130° for $\simeq 2$ hr. to sublime the excess of 8-hydroxyquinaldine, which would otherwise cause loss of Cu during the elution of the Cr by washing the column with 25 ml of benzene - $CHCl_3$ (1 + 1). If $<0{\cdot}05$ mg of Cr is present, the procedure for its separation can be omitted. The error varies from 0·5 to 2·0%.
W. J. BAKER

2096. **Dialkyl and diaryl phosphorodithioates as analytical reagents. I. Potentiometric determination of copper by means of nickel diethyl dithiophosphate.** A. I. Busev and M. I. Ivanyutin (M.V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1956, **11** (5), 523–528.—The use of OO-diethyl hydrogen phosphorodithioate $(C_2H_5O)_2PSSH$ (**I**) (*cf.* Busev, *Zhur. Anal. Khim.*, 1949, **4**, 49 and 234) as a precipitant for heavy-metal ions is studied. The nickel salt of **I** is synthesised, its reaction with Cu^{2+} is examined and the results are used in establishing a potentiometric method for determining Cu in several alloys and ores. It reacts with $CuSO_4$ according to the equation—

$$CuSO_4 + [(C_2H_5O)_2PSS]_2Ni = [(C_2H_5O)_2PSS]_2Cu + NiSO_4.$$

A further reaction occurs, but this does not affect the molar ratio of the reactants. Potentiometric titration of $CuSO_4$ is carried out with a mercury indicator electrode. Interference of Fe^{3+} is prevented by addition of H_3PO_4 and that of Sn^{2+} by oxidation to Sn^{4+}. To determine Cu in brass and other copper alloys and in ores, the material is dissolved in HCl, HNO_3 or H_2SO_4, oxides of N are removed if HNO_3 has been used, and the Cu is titrated with the nickel salt of **I** in the presence of any SiO_2 that may be there. With alloys containing Pb, the Pb is first removed as $PbSO_4$. Brass containing 59·84% of Cu gave a result of 60·09%, beryllium bronze with 97·26% gave 97·16%, and a sulphide ore containing 2·80% gave a result of 2·75% of Cu.
G. S. SMITH

2097. **Determination and separation of copper in metallurgical products.** E. C. Stathis (Athens Univ., Greece). *Anal. Chim. Acta*, 1957, **16** (1), 21–24 (in English).—Ascorbic acid is proposed as reducing agent in the pptn. of Cu as CuSCN, and methods are given for the determination of Cu by this means in silver alloys, bronzes, cast iron and steel.
W. C. JOHNSON

2098. **The colorimetric determination of copper in the presence of large quantities of gold.** V. F. Barkovskiĭ and M. S. Usova. *Izv. Sektora Platiny, I.O.N.Kh., Akad. Nauk, SSSR*, 1955, (32), 75–79; *Ref. Zhur., Khim.*, 1956, Abstr. No. 51,104.—The photocolorimetric determination of Cu in gold alloys in the presence of considerable quantities of Ni is based on the measurement of the intensity of the colour of the Cu - aq. NH_3 complex; pptn. of Au is prevented by addition of $Na_2S_2O_3$; Cu^+ are oxidised to Cu^{2+} by H_2O_2. Dissolve the sample of alloy (0·5 g) in aqua regia (15 ml), evaporate down to 2 ml, then evaporate with three 5-ml portions of water to a final vol. of 3 ml. Dilute with 20 ml of water, add freshly prepared 50% $Na_2S_2O_3$ soln. (15 ml), conc. aq. NH_3 (30 ml) and 3% H_2O_2 (1 ml), filter, make up to 100 ml and measure the extinction with a red filter in a 5-cm cell. Prepare a comparison soln. in the same way from a standard soln. of Ni containing the same amount of Ni as the soln. being analysed. Testing of the method on artificial mixtures has shown that the abs. error in the determination of Cu is $< \pm 0{\cdot}02\%$.
C. D. KOPKIN

2099. **New rapid and precise method of separating and estimating copper in the presence of iron and aluminium, and the further estimation of these elements.** G. Spacu and E. Rădulescu (Academ. R.P.R., Bucharest). *Stud. Cercet. Chim., Bucharest*, 1955, **3** (1–2), 7–11.—The weakly acid soln. containing Fe, Al and Cu is treated with Na sulphosalicylate to complex the Fe and Al. On the addition of pyridine and NH_4SCN, the Cu is pptd. as $[CuPy_2(SCN)_2]$ and is dried in a vacuum desiccator as described by Spacu and Dick (*Z. anal. Chem.*, 1939, **116**, 119; 1927, **71**, 185). The Fe and Al in the filtrate can then be determined by the usual methods.
J. H. WATON

2100. **The photocolorimetric determination of copper in platinum - copper and palladium - silver - copper alloys.** M. A. Chentsova, V. G. Levian and M. P. Yufa. *Izv. Sektora Platiny, I.O.N.Kh., Akad. Nauk, SSSR*, 1955, (32), 59–60; *Ref. Zhur., Khim.*, 1956, Abstr. No. 51,107.—Copper is determined by the intensity of the colour of the Cu - aq. NH_3 complex; Pd and Ag do not interfere; Pt is decolorised by the action of $NaHSO_3$ at pH 5. To analyse the Pt - Cu alloys, dissolve the sample

(0·3 g) in aqua regia (20 ml), evaporate to 0·5 ml and dilute to 10 ml; add 25% $NaHSO_3$ soln. (10 ml), and after 10 min. add 25% aq. NH_3 (20 ml); filter, dilute the filtrate to 100 ml and measure the extinction with a red filter in a 5-cm cell. A calibration curve is used for 1 to 6% of Cu, and a comparison method for higher percentages. To analyse Pd - Ag - Cu alloys, dissolve the sample (0·1 g) in 50% HNO_3 (10 ml), cool, add aq. NH_3 (20 ml), filter, dilute the filtrate to 50 ml and measure the extinction. The relative error is 3%.

C. D. KOPKIN

2101. **The determination of copper and silver by homogeneous precipitation with azimidobenzene [1H-benzotriazole].** N. I. Tarasevich. *Vestnik Moscov. Univ.*, 1955, (10), 111–113; *Ref. Zhur., Khim.*, 1956, Abstr. No. 51,106.—In determining Cu and Ag as described earlier (*cf.* Curtis, *Ind. Eng. Chem., Anal. Ed.*, 1941, **13**, 349), it is proposed to obtain 1H-benzotriazole (**I**) from *o*-phenylenediamine (**II**) during the course of the analysis by the action of $NaNO_2$ on **II** in acetic acid. Copper and silver are pptd. as $Cu(C_6H_4N_3)_2$ and $Ag(C_6H_4N_3)$ at pH 4·4 to 7. To the neutral soln. of Cu or the slightly acid (to methyl orange) soln. of $AgNO_3$ add a slight excess of a 1 to 2% soln. of **II**, crystalline $NaNO_2$ (3 moles per mole of **II**) and acidify with 30% acetic acid. After 15 to 20 min. warm the soln. to between 70° and 80°, then cool. After 1 hr., filter off the ppt., wash it with 1% acetic acid and dry at 120° to 150°. The conversion factors are, for Cu 0·21204, and for Ag 0·47734. The error is $\pm$1·42%. Cadmium does not interfere. C. D. KOPKIN

2102. **Photometric determination of copper and nickel in copper ores and metallurgical products by means of thiosemicarbazide.** J. Kinnunen and B. Merikanto (Outokumpu Oy, Metalworks, Pori, Finland). *Chemist Analyst*, 1956, **45** (4), 103–105.—The procedures described are based on the relative stabilities of the copper and nickel complexes of thiosemicarbazide in acid soln., Cu being determined in strongly acid media and Ni in weakly acid media in the presence of thiourea which decolorises the copper complex. Interfering elements are, *e.g.*, Fe^{III}, Ag, Cd, Zn and Co. The extinctions of the soln. are measured at 570 mμ for the copper complex and at 585 (or 910) mμ for the nickel complex. The results for copper ores agree well with those obtained by the electrolytic method. The procedure for Ni is recommended for use only with samples containing >5% of Ni and having a Ni to Cu ratio of at least 2:1, or with samples from which most of the Cu has been removed. O. M. WHITTON

2103. **The use of polarography and amperometric titration for determining gold.** N. A. Ezerskaya. *Izv. Sektora Platiny, I.O.N.Kh., Akad. Nauk, SSSR*, 1955, (32), 38–51; *Ref. Zhur., Khim.*, 1956, Abstr. No. 51,109.—To determine Au polarographically when in cyanide soln., first separate the Au by boiling with a 10% soln. of hydrazine hydrochloride, dissolve the ignited ppt. in aqua regia and evaporate the soln. to dryness with HCl. To the soln. of $HAuCl_4$ so obtained, add KOH and gelatin, pass H and polarograph. To determine Au in assay samples, dissolve in aqua regia, evaporate with a few drops of HCl and treat as above. For samples of slimes, treat with HNO_3 to remove Cu, dissolve in aqua regia, evaporate, ignite the residue, dissolve in HCl and again evaporate. The best background for the polarography is 2·5 *N* KOH or NaOH containing 0·02% of gelatin, when a wave is obtained at −0·9 V. The proportionality between the height of the wave and the concn. of Au is retained within wide concn. limits. Gold may also be estimated by amperometric titration with the rotating platinum electrode. As titrating soln., $Na_2S_2O_3$ is recommended for >0·1 mg of Au and mercaptobenzothiazole for >0·01 mg of Au. C. D. KOPKIN

2104. **Naphthazarin—a reagent for the colorimetric determination of beryllium.** G. G. Karanovich. *Trudy Vses. Nauch. Inst. Khim. Reaktivov*, 1956, (21), 43–47; *Ref. Zhur., Khim.*, 1956, Abstr. No. 54,747.—Naphthazarin (5:6-dihydroxy-1:4-naphthaquinone) (**I**) shows a colour transition from red through violet to blue over the pH range 6·8 to 8·9. It gives a colour reaction with Be. In an acetate buffer soln. at pH 5·6, 0·2 μg of Be in 5 ml of soln. can be determined by means of an aq. soln. of **I**; the maximum colour intensity is attained after 10 to 15 min. and is stable for 25 to 30 min. The complex of **I** with Be in acetone is stable for 18 hr. No interference is caused by Cd, Pb, Mn^{2+}, As^{5+}, Mo^{6+}, Ca, Mg, Ba, W^{6+}, Hg^{2+} and Na at ratios between 50:1 and 5000:1. Interference of >5 μg of Fe^{3+} is masked by tartaric acid (for 50 μg of Fe, 0·2 ml of 25% tartaric acid soln. and a 25% soln. of Na acetate of pH 5·6 are used). Cobalt, Ni and Zn are masked similarly, Cu^{2+} by means of SCN^- followed by $Na_2S_2O_3$, and Al by means of tartrate or citrate. To determine Be in alkali-metal salts, 1 to 2 ml of soln. (0·1 g of the salt) is diluted to 3 ml with the acetate buffer soln. and 1 ml of acetone together with 1 ml of a 0·01% soln. of **I** in acetone are added, and after 5 min. the extinction at 580 mμ is measured or the colour is compared with a scale of standards. G. S. SMITH

2105. **Colorimetric determination of small amounts of beryllium by means of organic reagents.** P. I. Vasil'ev. *Sb. Nauch. Tekhn. Inform. Ministerstvo Geol. i Okhrany Nedr*, 1955, (1), 131; *Ref. Zhur., Khim.*, 1956, Abstr. No. 54,748.—To determine Be (0·001 to 0·02% of BeO) in minerals, resorcinol is used, Al is masked by EDTA (disodium salt) and the interference of other elements is prevented by the extraction of the Be with a soln. of NaOH and Na_2S. The error is $\pm$0·002% of the sample wt. For rapid determination of Be in ores, quinalizarin is used, bivalent metals are masked by EDTA (disodium salt) and the colour is stabilised by means of Na_2S. Visual comparison of the colour with standards or in a photocolorimeter can be used in either method. G. S. SMITH

2106. **A gas-volumetric apparatus for the determination of milligram quantities of alkaline-earth metals.** P. S. Rogers, D. J. M. Bevan and J. W. Tomlinson (Imperial College, London, England). *Mikrochim. Acta*, 1956, (12), 1839–1841 (in English).—An apparatus for the determination of alkaline-earth metals dissolved in, or mixed with, a salt by measuring the volume of hydrogen which is evolved when the sample reacts with water is described. An accuracy better than $\pm$1% is claimed. Errors due to desorption of gases by glass during the reaction are avoided. P. S. STROSS

2107. **Paper chromatography of inorganic compounds. I. Alkali and alkaline-earth chlorides.** I. Vavruch and M. Hejtmánek (Dept. of Phys. Chem., Inst. of Chem. Technol., Prague). *Coll. Czech. Chem. Commun.*, 1956, **21** (6), 1363–1375 (in English).—In the paper chromatography of inorganic ions, influential factors are the selective

extraction of salts by the solvents used, the adsorption of ions on the paper, and the formation of complexes with the solvents. Methanol (97%), ethanol (98%), *n*-propanol, *n*-butanol, *n*-pentanol, acetone, pyridine and benzene were examined. The only salts examined were chlorides. These were detected by spraying the paper with 0·5 *N* $AgNO_3$ and exposing to daylight until greyish-black spots developed. R_F values of Li^+, K^+, Ca^{2+} and Cu^{2+}, and solubilities in various solvents are tabulated. If an increasing amount of solvent in which the salt shows high mobility is added to a solvent in which it is immobile, the R_F increases. The duration of analysis, *i.e.*, the distance of the solvent from the origin at the end of the analysis, had a significant influence on R_F values. The dependence of the height of the salt band on concn. was studied with the chlorides of K, Na and Li, partly with respect to its possible application in the simultaneous determination of Na and K. The band height increases with increasing concn., but dependence is not linear; an equation is suggested. Conductimetric studies were made to detect the relative positions of individual salts on the paper after chromatographic analysis. Distribution of KCl - NaCl - LiCl on the chromatogram is shown diagrammatically. Adsorption from alcoholic soln. is always higher than from aq. soln.

W. J. WRIGHT

2108. **An amperometric method for magnesium.** J. A. Dean and H. A. Bryan (The Univ., Knoxville, Tenn., U.S.A.). *Anal. Chim. Acta*, 1957, **16** (2), 180–185 (in English).—Milligram amounts of Mg can be determined amperometrically with an error of $\simeq 1\%$, by utilising the polarographic wave given by the Mg complex of Pontachrome violet SW (*Ibid.*, 1957, **16**, 87). The titration is made at pH 11 and −0·60 V *vs.* the S.C.E. in the presence of 1·2 *M* ethylenediamine to minimise interference from Ca and small amounts of other cations. The pH should be adjusted to within 0·1 with piperidine - HCl buffer. The accuracy is highest between 0·001 and 0·01 *M* concn. of Mg, and results are satisfactory even at concn. of 3×10^{-4} *M*. No preliminary separation is needed for most samples of limestone, otherwise the titration is made on the filtrate from the R_2O_3 ppt.

W. J. BAKER

2109. **Spectrophotometric determination of magnesium with 1-azo-2-hydroxy-3-(2:4-dimethylcarboxanilido)naphthalene-1′-(2-hydroxybenzene)[3-hydroxy-4-o-hydroxyphenylazonaphth-2′:4′-xylidide].** C. K. Mann and J. H. Yoe (The Univ., Austin, Texas, U.S.A.). *Anal. Chim. Acta*, 1957, **16** (2), 155–160 (in English).—From 0·02 to 0·4 p.p.m. of Mg in 25 ml of soln. can be determined spectrophotometrically at 505 mμ (1-cm cell) by utilising the coloured complex of Mg with the reagent in ethanolic soln. and in the presence of 0·08 *M* aq. $Na_2B_4O_7$ as buffer. The aliquot used for the determination should contain from 1 to 10 μg of Mg; the colour is developed immediately. Optimum operating conditions, accuracy, sensitivity ($\simeq 1$ in 5×10^7), and possible interference by other ions are discussed. Provided that the content of H_2O in the soln. is $\simeq 30\%$, and only Cl^- and borate are present, $\not> 20$ p.p.m. of Ca or Sr can be tolerated.

W. J. BAKER

2110. **Spectrographic determination of calcium as impurity in ferrous sulphate.** G. A. Pevtsov. *Trudy Vses. Nauch. Inst. Khim. Reaktivov*, 1956, (21), 64–65; *Ref. Zhur., Khim.*, 1956, Abstr. No. 54,750.—The spectrum is excited by an activated a.c. arc (200 V, 3·5 amp.) with a pre-spark of 2 to 3 min. at 6 amp. With traces of Ca, the lines Ca 3933·67, 3968·47 and 4226·73 A are measured. With the first, equivalence of intensity with the lines of Fe 3906·48, 3920·26 and 3902·94 A corresponds, respectively, with contents of Ca of 0·004, 0·012 and 0·040%.

G. S. SMITH

2111. **Proposed method of test for calcium and magnesium oxides in lime.** American Society for Testing Materials (1916 Race Street, Philadelphia, Pa., U.S.A.). *Bull. A.S.T.M.*, 1956, (218), 27–28.—The Ca in the sample soln. is titrated first with EDTA (murexide - NaCl indicator), and then the Ca plus Mg is titrated on a fresh aliquot (Eriochrome black T indicator) in the presence of NH_4Cl - aq. NH_3 buffer and, if Ba and Sr are present, of 3 ml of saturated aq. K_2SO_4. The method is applicable even to samples high in R_2O_3 or Mg or both.

W. J. BAKER

2112. **Note on a method of staining to distinguish between calcite and dolomite.** J. Mitchell (Geol. Survey Dept., Gold Coast). *Colon. Geol. Miner. Res.*, 1956, **6** (2), 182.—Sodium alizarinsulphonate (alizarin red S) (0·1% in *N*/15 HCl) was found satisfactory. Calcite forms a transparent reddish-violet lake, whilst dolomite is unaffected.

N.E.

2113. **Indirect colorimetric method for the determination of barium. III. Determination of barium in silicate rocks.** Tôru Nozaki (Fac. Engng, Ehime Univ., Niihama). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1956, **77** (9), 1430–1432.—Barium (0·5 to 2 mg) is pptd. as $BaSO_4$, which is treated by heating with water (20 to 30 ml) and Amberlite IR-120 (H) (1 g) at 80° for 3 hr. The Ba on the resin is eluted with 2 *N* HCl (130 ml), the eluate is evaporated to a vol. of 1 to 2 ml and treated with $(NH_4)_2MoO_4$ to precipitate $BaMoO_4$; the ppt. is dissolved and the Mo is colorimetrically determined with KSCN (*cf. Anal. Abstr.*, 1955, **2**, 1160). In the presence of Ca >50 times the weight of the Ba, $BaSO_4$ contaminated by $CaSO_4$ is repptd., and the last ppt. is treated as described above. Rock samples (1 to 2 g) are fused with Na_2CO_3 ($\simeq 5$ times the weight of the sample) and Ba is pptd. as $BaSO_4$.

K. SAITO

2114. **Determination of microgram quantities of barium in sodium.** D. Dutina (Knolls Atomic Power Lab., Schenectady, N.Y., U.S.A.). *U.S. Atomic Energy Comm.* KAPL–1425, 1955, 19 pp.—Barium is pptd. as $BaCrO_4$, followed by spectrophotometric determination of the CrO_4^{2-}; 250 p.p.m. of Ba can be determined by this method.

CHEM. ABSTR.

2115. **Co-precipitation of radium with barium sulphate.** L. Gordon and K. Rowley (Univ. Syracuse, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 34–37.—The co-pptn. of Ra with $BaSO_4$ was studied by pptn. from homogeneous soln. by the hydrolysis of sulphamic acid. A combined reaction and centrifugation cell effected complete separation of solid and liquid phases, and enabled accurate measurements to be made of the distribution of the radioactive tracer between the two phases. The results conform to the Doerner - Hoskins equation, the distribution coeff. (λ) being const., within experimental error, over the range in which the fraction (f) of the Ba pptd. is 0·03 to 0·96. At 90° the value of λ is $1{\cdot}21 + 0{\cdot}009\,f$; between temp. ($T$)

of 50° and 90° it can be calculated from $\log_{10} \lambda = 220/(273 + T) - 0{\cdot}520$. W. J. BAKER

2116. **Thioacetamide in the separation and EDTA titration of zinc in aluminium alloys.** A. M. Amin (Chem. Engng Dept., Univ. Cairo, Giza, Egypt). *Chemist Analyst*, 1956, **45** (4), 95, 101–102.—To a sample of 1 to 2 g, 5 ml of H_2O and 20 ml of 15% NaOH soln. are added. After evolution of H ceases, 50 ml of hot H_2O are added and the mixture is boiled and filtered. The residue is washed and the combined filtrates are boiled with 20 ml of thioacetamide soln., 4% (w/v), for 3 min. The pptd. ZnS is washed and dissolved in hot HCl (1:1). The filtrate is boiled to expel H_2S, cooled, and made up to 250 ml. To 20 ml are added aq. NH_3 until the soln. is slightly alkaline, buffer soln. to make the pH of the soln. 10, about 10 mg of ascorbic acid, 1 ml of 10% soln. of KCN to mask other metals, 1 ml of 30% triethanolamine soln. (or NH_4F), and about 3 mg of Eriochrome black T indicator powder. If the soln. be coloured violet, it is titrated with 0·1 *M* EDTA (disodium salt) until a blue colour appears, and the volume of EDTA soln. corresponds to the sum of Mn and Mg. Then, or if the colour were initially blue, the Zn is selectively demasked by addition of 5% formaldehyde soln. until the soln. is violet. The soln. is then titrated in the cold with EDTA (disodium salt) to a blue colour. If on addition of a further drop of formaldehyde soln. the violet colour returns, the EDTA titration is continued. The operation is repeated until a permanent blue colour is obtained. The volume of EDTA soln. required after demasking the Zn corresponds to the Zn only. The determination of Zn in six samples takes about 3 hr.

O. M. WHITTON

2117. **Conditions of the separation of zinc and cobalt from aluminium and iron by the method of labelled atoms.** Yu. V. Morachevskiĭ and Z. S. Bashun (Inst. Chem. of Silicates, Acad. Sci., U.S.S.R.). *Izv. Akad. Nauk. SSSR*, 1956, (10), 1185–1196.—Three methods, pptn. with aq. NH_3, the 'basic acetate' separation, and pptn. with pyridine, for separating Al and Fe from Zn and Co are studied by the aid of ^{65}Zn and ^{60}Co. With aq. NH_3, complete separation of Zn and Co is attained only in a narrow pH range, 5·5 to 5·9, and in the presence of $\leq$5% of an ammonium salt. Under these conditions the separation is not affected by the ratio of the amounts of the ions. Iron is a collector for Al and its presence permits complete pptn. of Al at relatively low pH values. With the 'basic acetate' method, the presence of Fe in amount exceeding that of Al is essential for the complete separation of Al from Zn and Co. The pyridine method gives an excellent separation of Al and Fe from Zn and Co. G. S. SMITH

2118. **Microgram estimation of cadmium and zinc by EDTA titration after paper-chromatographic separation.** I. I. M. Elbeih and A. M. Amin (Chem. Dept., Univ. Cairo, Giza, Egypt). *Chemist Analyst*, 1956, **45** (4), 94–95.—Cadmium and zinc are separated from their nitrate soln. in 0·1 *N* HNO_3 by paper chromatography of a sample soln. (0·01 ml); their location on the chromatogram is detected by treatment with oxine and exposure to u.v. light in the presence of NH_3. After extraction from the paper, by boiling with 15 ml of H_2O and 1 ml of buffer soln. (13·5 g of NH_4Cl and 88 ml of conc. aq. NH_3 diluted to 250 ml with H_2O), and cooling the soln. to about 30°, 1 ml more of buffer soln., about 5 mg of ascorbic acid (to obviate interference by Fe^{III}), three drops of 5% KCN soln. (to mask heavy metals), and about 2 mg of Eriochrome black T indicator powder are added. To selectively demask Zn and Cd, 3% formaldehyde soln. is added dropwise until a violet colour appears. The soln. is then titrated with 0·001 *M* EDTA (disodium salt) until the colour changes to blue. One drop of formaldehyde is added and, if the colour changes to violet, the titration with EDTA soln. is continued. The formaldehyde addition and EDTA titrated are repeated until one drop of formaldehyde does not affect the blue colour. The procedure is applied to four blank paper pieces and four spots of each metal. With amounts of <25 μg of Cd or 10 μg of Zn, results were unsatisfactory. However, in such cases titration of two or more spots together gave satisfactory results, of max. error $\pm 1{\cdot}1$ μg of Cd and 1·3 μg of Zn. If the soln. contains $>1\%$ of each metal, the spots will be overloaded.

O. M. WHITTON

2119. **Oxidation of 2-*o*-hydroxyphenylbenzoxazole with potassium permanganate and its application to volumetric determination of cadmium.** Yoshio Horiuchi and Minoru Saito (Fac. Engng, Iwate Univ., Morioka). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1956, **77** (9), 1340–1344.—The oxidation of 2-*o*-hydroxyphenylbenzoxazole (**I**) with $KMnO_4$ was studied in 14% H_2SO_4 and it was found that one mole of **I** reduces 50 equiv. of O. Since **I** produces ppt. with Cd or Cu in a basic soln. of pH 11, an indirect volumetric determination of Cd was effected. The soln. of Cd (<50 mg) is adjusted to pH 11 with NaOH and tartaric acid, and treated with a slight excess of 1% **I** soln. at 60° for 30 min. The ppt. is washed with ammoniacal 50% ethanol, then dissolved in 28% H_2SO_4 and diluted to 100 ml. A portion is treated with 0·1 *N* $KMnO_4$ ($\simeq 1{\cdot}5$ times the equiv. of **I**) at 22° to 24° for 1 hr., and the excess of the $KMnO_4$ is determined iodimetrically with 0·1 *N* $Na_2S_2O_3$. The presence of Ni, Co, Zn and Hg vitiates the estimation. K. SAITO

2120. **Determination of micro quantities of cadmium in uranium.** R. H. Rodríguez Pasqués and J. F. Possidoni de Albinati (Anal. Chem. Admin., National Atomic Energy Div., Argentina). *An. Asoc. Quim. Argentina*, 1956, **44** (2), 90–103.—The effect of variables on the determination of Cd by spectrophotometry at 520 mμ of the compound formed with dithizone is studied; Zn and Tl do not interfere. The dithizone concn. influences the results and a value of 0·004% is recommended. The strength of the light and the concn. of U do not appreciably affect the results, but the colour fades after a time and it is recommended that the light transmission be measured within 5 min. of its development. It was found advisable to wash the CCl_4 solvent with NaOH before use.

D. LEIGHTON

2121. **Tetraethylthiuram disulphide [disulfiram] as an analytical reagent. IV. The photometric determination of mercury and silver.** J. Michal and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (1), 56–62.—Disulfiram (Dicupral) (**I**) reacts with Cu^{2+} to form a yellow-brown complex (*Anal. Abstr.*, 1955, **2**, 2040, 2345), which is decomposed and decolorised by an exchange reaction with Hg^{2+} and Ag^{+}. This reaction was used for the indirect photometric determination of Hg and Ag. *Reagent*—Mix equal vol. of a satd. soln. of $CuSO_4$ and **I** in

ethanol (50%) and set aside for 48 hr. Filter off and dry the crystals; the name Mercupral was proposed for this compound (**II**). A soln. of 10 mg of **II** in benzene (250 ml) was used as the reagent. *Procedure*—To the sample containing Hg (10 to 50 μg) or Ag (20 to 160 μg) in a separating funnel add soln. of **II** (2·00 ml) and benzene (15 ml) and shake for 1 min. (Ag) or for 3 min. (Hg). Separate the benzene layer into a volumetric flask (25 ml), dilute to vol. with ethanol (95%) and mix. Measure the decreased extinction at 445 mμ. Compare with a calibration curve. Excess of Al^{3+}, As^{V}, Sn^{IV}, Ca^{2+}, Mg^{2+}, Zn^{2+}, Fe^{3+}, Ti^{IV}, W^{VI}, Pb^{2+}, Mn^{2+}, Co^{2+}, Mo^{VI}, Cd^{2+}, Tl^{+}, Ni^{2+}, Ba^{2+}, Sb^{3+}, Cr^{3+}, Bi^{3+}, V^{V} and Cu^{2+} does not interfere; HNO_3 must be absent. J. ZÝKA

2122. **Separations involving sulphides. II. Separation of arsenic, molybdenum and tellurium from mercury.** G. B. S. Salaria (Univ. of Allahabad, India). *Anal. Chim. Acta*, 1957, **16** (1), 3–5 (in English).—A soln. containing Hg^{2+}, AsO_4^{3-} and sufficient HCl to dissolve the mercuric arsenate is treated with a large excess of 2 *N* Na_2S (*cf. Anal. Chim. Acta*, 1954, **13**, 28) and the thio salt of Hg is decomp. by shaking with an excess of ammonium acetate. The HgS is filtered off in a sintered-glass crucible (porosity **4**), washed with water, alcohol and ether, dried at 105° and weighed. Another portion of the soln. is treated with 2 *N* Na_2S and then made 6 *N* with respect to HCl; the mixture is heated to boiling-point, allowed to cool and the mixture of HgS and As_2S_5 is filtered off, dried and weighed as in the first experiment. The wt. of As_2S_5 is obtained by difference. A similar procedure is used to determine Hg and MoO_4^{2-}, but the mixed thio salts are decomp. in *N* HCl and the MoS_3 is dried in a vacuum desiccator (*cf. Anal. Abstr.*, 1954, **1**, 220). In the determination of Hg and TeO_3^{2-}, the decomp. occurs in *N* HCl and the TeS_2 is dried at 105°. W. C. JOHNSON

2123. **Application of the aluminium indicator electrode to the determination of aluminium by the fluoride method.** E. N. Ovsepyan and V. M. Tarayan. *Nauch. Trudy Erevan Univ.*, 1956, **53**, 85–94; *Ref. Zhur., Khim.*, 1956, Abstr. No. 54,755.—In a study of the application of the method of Chirkov (*Zavod. Lab.*, 1948, **14**, 783) a nichrome wire is used as a comparison electrode. In the titration of Al, the amount of 0·5 *N* NaF is less than that required for the formation of the insoluble compound $3NaF.AlF_3$. By saturation of the soln. of Al with NaCl, the compound $11NaF.4AlF_3$ is pptd., for which the optimum pH range is 3 to 7·3. With the compensation method and the use of a S.C.E., the electrode potential of the aluminium electrode in a soln. of Al with an acetate buffer soln. is $\simeq -1{\cdot}1$ V. In the titration with NaF, the potential at the end-point is $-1{\cdot}38$ V. The potential of the aluminium electrode remains const. when titration of Al is carried out by means of a soln. of 8-hydroxyquinoline. It is considered that F^- attack the protective film on the aluminium electrode and thus change its potential. G. S. SMITH

2124. **Sources of error in microchemical aluminium determinations through leaching from glass and impurities in chemicals.** W. Oelschläger (Landw. Hochschule, Stuttgart, Hohenheim, Germany). *Z. anal. Chem.*, 1957, **154** (5), 329–333.—A study has been made of the amounts of Al and Fe that can be leached from glassware by acids and alkalis or are present as impurities in various reagents. With glassware it was found that as much as 125 μg of Al was removed by heating 30 ml of 0·9 *N* NaOH soln. in a vessel for 60 min. Aq. NH_3 soln. (0·9 *N*) removed as much as 16 μg after heating for 60 min. and allowing to stand for a further 1 hr. Under similar conditions HCl and H_2SO_4 (0·3 and 0·9 *N*) removed <1 μg of Al. These amounts, whilst insignificant in macro-analysis, are a likely source of error in micro-determinations. Quantities of Fe leached from glassware were of a much lower order (0·3 to 7 μg). Examples of Al (per 100 g) found in designated analytical reagents were—$FeCl_3.6H_2O$, 4000 μg; NaOH, 1400 μg; conc. HCl, 250 μg; conc. aq. NH_3, 83 μg; and dist. H_2O, 2 μg. Iron (per 100 g) varied from NaOH, 230 μg, to conc. HNO_3, 28 μg. D. F. PHILLIPS

2125. **Rapid colorimetric method for the determination of aluminium in copper - aluminium alloys.** M. C. Steele and L. J. England (Metal Mfg. Ltd., Port Kembla, Australia). *Anal. Chim. Acta*, 1957, **16** (2), 148–149 (in English).—Aluminium in aluminium-brass and -bronze can be determined to within 0·01% on a 1-g sample dissolved in HNO_3 (10 ml). *Procedure*—Dilute the soln. to 100 ml and to a 10-ml aliquot add 20% aq. NaOH (5 ml), boil for 1 to 2 min. and dilute to 100 ml. Allow the ppt. to settle, filter if necessary, and to a 5-ml aliquot of the filtrate add fresh 0·005% aq. Eriochrome cyanine (20 $\pm$ 0·5 ml) followed by a few drops of glacial acetic acid until the colour is a clear red. Dilute to 100 ml and, after exactly 15 min., measure the extinction at 550 mμ. A blank containing 1 g of Cu must be run in each series of determinations. Lead, Zn, Sn and Ni do not interfere; the treatment with NaOH controls interference from Fe, Mg, Cu or As. W. J. BAKER

2126. **Polarographic determination of aluminium in zinc-base die-casting alloys.** J. A. Page, D. H. Simpson and R. P. Graham (McMaster Univ., Hamilton, Canada). *Anal. Chim. Acta*, 1957, **16** (2), 194–200 (in English).—In the procedure given, interfering elements (Zn, Cu, Pb, Cd and others) are first removed by electrolysis of the sample soln. for 1 hr. in a mercury-cathode cell at 5 to 9 V and 5 amp. The aluminium ($\simeq$4%) is retained quant. in the electrolyte, with only $\simeq$0·03% of Zn, which does not interfere subsequently. The soln. is then diluted and an aliquot is polarographed at 25° and pH 3·65 $\pm$ 0·03 (control of this value is critical) in a supporting electrolyte of 0·15 *M* $MgCl_2$ and 0·045 *M* Na_2SO_4 at a potential of $-1{\cdot}0$ to $-2{\cdot}0$ V *vs.* the S.C.E. The precision and accuracy are satisfactory for routine analyses. W. J. BAKER

2127. **Spectrophotometric determination of aluminium in titanium and titanium alloys. An aluminon method.** D. K. Banerjee (Chase Brass & Copper Co., Waterbury, Conn., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 55–60.—In the rapid routine procedure described, an aliquot of the sample soln. containing $>$20 mg of Ti is taken. Titanium, Fe, W, Mo, V, Sn, Sb and Bi are removed by a rapid cupferron pptn. (*cf. Ibid.*, 1953, **25**, 1437), the excess of cupferron in the filtrate is oxidised, any Cr is removed by volatilisation, and the Al in the residual soln. is then determined spectrophotometrically with aluminon in the presence of $HClO_4$ and 1% thioglycollic acid (the latter reagent is omitted if much Co is present). The colour is

developed for ≃30 min. at 100°, and the extinction of the cool soln. is measured at 540 mμ in 1-cm cells, against H_2O for low concn. and (by differential spectrophotometry) against a standard soln. (100 μg of Al) for high concn. (1 to 10%). At the high concn. the aliquot should contain from 100 to 150 μg of Al, but at concn. of 0·1 to 1% it should contain 20 to 30 μg of Al. The content of Al in the aliquot is calculated from calibration curves. Precision and accuracy are good; the standard deviation varies from ±0·01 to 0·1 for concn. of Al of 2 to 10% (differential colorimetry). Variables and the effect of possible interferences are discussed. W. J. BAKER

2128. **The solubility of aluminium and titanium 8-hydroxyquinolinates in the presence of complexing reagents, and the separation of these elements by 8-hydroxyquinoline.** I. V. Pyatnitskiĭ. *Nauk. Zap. Kiev Univ.*, 1955, **14** (4), 131–141; *Ref. Zhur., Khim.*, 1956, Abstr. No. 47,238.—The solubility of the 8-hydroxyquinolinates of Al and Ti in acetic and oxalic acids in relation to the pH and excess of precipitant is studied. To determine Ti in the presence of Al (≯0·01 *M*), add to the soln. oxalic acid to 0·25 *M*, 2 *N* aq. NH_3 to a blue colour with bromocresol purple (pH 6), and a 2% ethanolic soln. of 8-hydroxyquinoline (9 to 10 ml in excess); boil for 10 min., and set aside overnight. Filter off the pptd. titanium complex, wash it with hot water, then dissolve it on the filter with conc. HCl and complete the determination by titration with bromate. C. D. KOPKIN

2129. **A spectrographic technique for the determination of gallium in micro samples.** E. M. Murt (Philco Corp., Lansdale, Pa., U.S.A.). *Appl. Spectroscopy*, 1956, **10** (4), 210–213.—A technique to enhance the sensitivity of the determination of Ga in micro samples of In - Ga alloys is described. The use of a helium atmosphere reduced the cyanogen band interference with the most sensitive Ga lines at 4172·1 and 4033·0 A, so that it becomes possible to detect 0·05 μg of Ga in a sample of 50 μg of alloy. The method could be extended to other elements which normally have their most sensitive lines masked by cyanogen bands. K. A. PROCTOR

2130. **New rapid method for the gravimetric estimation of thallium.** P. Spacu and G. Hlevca Bucharest, Romania). *Stud. Cercet. Chim., Bucharest*, 1955, **3** (3–4), 203–207.—Thallium[I] is pptd. quant. as the complex $Tl[Cr(SCN)_4(NH_3)_2]$ by the addition of a 3·5% aq. soln. of Reinecke's salt to an acid, neutral or feebly alkaline soln. of Tl^I. After filtration, the ppt. is washed with ethanol and with ether, and is dried in a vacuum desiccator. The estimation can be carried out in the presence of most common ions, but Pb interferes. The analysis requires 60 to 70 min. J. H. WATON

2131. **Colorimetric methods of determining small amounts of thallium.** N. T. Voskresenskaya (M.V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1956, **11** (5), 585–589.—The colour reaction between the anion $TlBr_4^-$ and methyl violet (*cf.* Gur'ev, *Nauch. Trudy Gintsvetmeta*, 1955, (10), 371) and brilliant green is studied. The soln. containing Tl is mixed with 1 to 4 ml of *N* HBr and 2 ml of bromine water, then diluted to 20 ml with water and boiled to remove the excess of Br. The soln. containing $TlBr_4^-$ is mixed with two drops of 0·01% methyl violet soln., diluted to 25 ml and extracted with 5 ml of amyl acetate. The extinction of the extract is measured with a green filter. With brilliant green, 2 to 4 ml of *N* HBr and 1 ml of 0·01% dye soln. and a red filter are used. The sensitivities are 0·05 and 0·03 μg per ml, respectively, Beer's law is obeyed over the concn. ranges 0·05 to 4·0 and 0·05 to 5·0 μg per ml, and the molar extinction coeff. are 53,000 and 70,000. The interference of a large number of ions can be avoided by means of known methods. G. S. SMITH

2132. **Possible losses of thallium in silicate analysis.** V. I. Kuznetsov and G. V. Myasoedova (V.I. Vernadskiĭ Inst. of Geochem. and Anal. Chem., Acad. Sci., USSR). *Zhur. Prikl. Khim.*, 1956, **29** (12), 1875–1876.—Fusion of silicates and other materials with Na_2CO_3 or with a mixture of Na_2CO_3 and K_2CO_3 results in almost complete volatilisation of any Tl present. G. S. SMITH

2133. **Chemistry of the rare elements. II. Scandium pyrophosphate.** F. Petrů, B. Hájek and J. Závorka (Inst. Inorg. Chem., High School Chem. Technol., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (1), 21–26.—Potentiometric, derivative potentiometric and conductimetric methods show that scandium salts, when treated with $Na_4P_2O_7$, form an insol. ppt., the composition of which (after ignition) corresponds to the formula $Sc_4(P_2O_7)_3$. J. ZÝKA

2134. **Electro-deposition of the actinide elements.** R. Ko (Hanford Atomics Products Operation, General Electric Co., Richland, Washington, U.S.A.). *Nucleonics*, 1957, **15** (1), 72–77.—The method previously described (*Anal. Abstr.*, 1957, **4**, 1479) for Am has been extended to the deposition of Th, U, Np, Pu and Cm. Thorium is quant. deposited in 16 hr. on Cu, with a current of 50 mA, from 0·05 *M* H_2SO_4 - 0·08 *M* ammonium formate, or in 4 hr. if the soln. contains 50% of ethanol. Uranium and Np are quant. deposited in 1 hr. on Cu, at 80 to 100 mA, from 0·2 *M* $HClO_4$ - 0·15 *M* ammonium formate; deposition occurs on Pt which has been treated by boiling in HNO_3 and then in HCl. An 80% yield on stainless steel was achieved when 1 μg of Pu in 0·2 *M* formic acid - 0·05 *M* ammonium formate was electrolysed at 100 mA for 1 hr.; with 10 μg the recovery was 90%; up to 115 μg has been deposited in 4 hr. at 50 mA from 0·05 *M* H_2SO_4 - 0·076 *M* ammonium formate with yields of 96%. Curium is deposited by using the same conditions as for Am. G. J. HUNTER

2135. **Fluorimetric analysis. II. Determination of yttrium with oxine.** Masayoshi Ishibashi, Tsunenobu Shigematsu and Yasuharu Nishikawa (Chem. Dept., Fac. Sci., Kyoto Univ., Sakyo-ku, Kyoto). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1956, **77** (10), 1474–1479.—Yttrium oxinate (**I**), pptd. from an ammonium acetate buffer of 9·5, gives an intense fluorescence in $CHCl_3$, which has a max. at 500 mμ. Its intensity decreases with time, but is proportional to amounts of **I** <50 μg at any given time. **I** appears to be a 1 to 1 addition compound of Y trioxinate and oxine, which readily dissociates at temp. higher than 100°. The extraction of **I** with $CHCl_3$ from aq. soln. of various pH values was studied with the use of radioactive ^{90}Y as tracer. The extraction reaches a maximum at pH 8·5 and remains unchanged with further increase in pH. Since the excess of oxine in the aq. layer increases the intensity of fluorescence in the $CHCl_3$ layer, the fluorescence is also ascribed to **I**. A similar fluorescence is given by La and Ce, but the

intensity is about one-tenth that of Y. The sample soln. (<50 μg of Y), eluted from a cationic resin column with dil. citric acid, is mixed with 1% oxine in *N* acetic acid (1 ml) and 20% ammonium acetate (5 ml) and made pH 9·5 with *N* aq. NH_3 soln. The product is extracted with $CHCl_3$ (3 × 10 ml), made up to 50 ml and filtered, and its fluorescence is measured at 500 mμ after 15 min., with a mercury lamp (365 mμ) as light source.

III. Determination of indium with 8-hydroxyquinaldine and oxine. Masayoshi Ishibashi, Tsunenobu Shigematsu and Yasuharu Nishikawa. *Ibid.*, 1956, **77** (10), 1479–1482.—Similar experiments were carried out with In oxinate (**II**) and 8-hydroxyquinaldate (**III**). The optimum pH values for their extraction with benzene are 5·1 to 8·2 and 7·0 to 8·5, respectively. Excess of the reagent is also extracted and causes a decrease in the intensity of fluorescence (max. 535 mμ and 520 mμ for **II** and **III**), which remains unchanged for 24 hr. Metal ions, including those of Cu, Ni, Fe, Zn, Be and Ga, vitiate the estimation. Interference is minimised by the use of **II** in benzene. The sample soln. is treated with oxine (0·5%, 1 ml) and ammonium acetate (20%, 2 ml) and adjusted to pH 8 with aq. NH_3 soln. The product is extracted with benzene (3 × 10 ml), the extracts are made up to 50 ml, and the fluorescence is similarly measured. The working curve is linear for 10 to 100 μg of In. K. Saito

2136. **Chromatographic separation of radioisotopes of elements of the yttrium group obtained by fission of ytterbium and hafnium by protons of high energy.** A. A. Pozdnyakov (V.I. Vernadskiĭ Inst. of Geochem. and Anal. Chem., Acad. Sci., U.S.S.R., Moscow). *Zhur. Anal. Khim.*, 1956, **11** (5), 566–571.—The products obtained by irradiation of 70 to 80 mg of Yb acetate or $Hf(OH)_4$ with high-energy protons were dissolved in 5% acetic acid (for Yb) or a mixture of 0·5 *N* HCl and 5% oxalic acid (for Hf). Separation of Yb was carried out by electrolysis with a mercury cathode, and that of Hf by passing the soln. through a column of resin (H form) which retained the rare-earth elements but not Hf. For separation of the radio-isotopes of the rare earths, a cationite (NH_4 form) at 90° was used with a soln. of 0·4 *M* ammonium lactate at exactly pH 3·4.

G. S. Smith

2137. **The position of yttrium in the exchanger separation of yttrium earths, where nitrilotriacetic acid is the eluent.** L. Holleck and L. Hartinger (Chem. Inst. der Hochschule, Bamberg). *Angew. Chem.*, 1956, **68** (12), 412.—The rare earths decrease in affinity for the exchanger and are more strongly complexed with increasing atomic number. As a result, yttrium earths must be separated at lower pH values than are cerium earths. They are merely enriched at pH values at which cerium earths may already be separated quantitatively. The occurrence of Y, together with the difficultly separable elements Er, Ho and Dy, may cause additional difficulties. During the present investigations of yttrium earth mixtures, the Y was unexpectedly found in the final fractions. A positional shift in the direction of the lighter rare-earth elements has already been noted in a number of classical separations. It has also been found that, depending on the pH value, the orders Dy, Tb, Y, and Dy, Y, Tb were obtained when citric acid was the complexing agent. When EDTA was used it was found that in this complex series Y was placed between Dy and Tb. Since earth mixtures used in these investigations contained no Eu or Sm, the position of Y under these conditions could not be accurately established. A test mixture to which Nd and Pr had been added, however, showed that Y lies between Gd and these. Positioning of Y between Gd and Eu would appear to be feasible. The analysis of the lanthanides was done spectrophotometrically by measuring the extinction at the maxima of the strongest absorption bands. L. S. Adler

2138. **A new reaction for the detection of Ce^{3+} in salts of Ce^{4+}.** L. M. Kul'berg and M. N. Ambrozhiĭ (Dept. Anal. Chem., Saratov Univ., SSSR). *Uch. Zap. Saratov Univ.*, 1955, **42**, 63–64; *Ref. Zhur., Khim.*, 1956, Abstr. No. 47,284.—The detection of > 3% of Ce^{3+} in preparations of Ce^{4+} is based on the formation of a grey - blue ppt. on the simultaneous pptn. of Ce^{3+} and UO_2^{2+} by aq. NH_3. To 1 ml of the soln. (containing ≃10 mg of cerium salt) add 3 to 5 drops of a satd. soln. of uranyl acetate and 4 to 5 drops of conc. aq. NH_3 and boil for 1 to 2 min. After 5 min. a blue ppt. is formed with <10% of Ce^{3+}, a grey ppt. with 5 to 8% of Ce^{3+}, and a grey - yellow ppt. with 3 to 4% of Ce^{3+}. With a small content of Ce^{3+}, a control experiment is carried out with salts of Ce^{4+}; the ppt. is pale yellow. The sensitivity of the reaction is > 100 μg of Ce^{3+}, with a limiting dilution of 1 in 1×10^4. C. D. Kopkin

2139. **Amperometric titration of cerium (III) with ferricyanide.** G. W. Leonard, H. J. Keily and D. N. Hume (Inst. Technol., Cambridge, Mass., U.S.A.). *Anal. Chim. Acta*, 1957, **16** (2), 185–188 (in English).—From 1 to 10 mg of Ce^{3+} can be determined accurately, even in the presence of a large excess of Ce^{4+}, by amperometric titration with 0·01 *M* $K_3Fe(CN)_6$ in the presence of 3 to 4 *M* K_2CO_3. A rotating platinum micro-electrode kept at the potential of the S.C.E. is used; oxygen must be rigorously excluded during the titration and from all the soln. used. The error is ≃ ±0·2%.

W. J. Baker

2140. **Ascorbimetric determination of quadrivalent cerium.** Z. Jach, J. Pačovský and M. Svach (Inst. anal. Chem., Montanhochschule, Ostrau, Czechoslovakia). *Z. anal. Chem.*, 1957, **154** (3), 185–187.—Ascorbic acid is used as a reducing agent in the determination of Ce. Variamine blue B hydrochloride is used as an indicator. The soln. is buffered with glycine - HCl - NaCl soln. and titrated with 0·01 *N* ascorbic acid in 0·1 *N* HCl until the violet - blue colour is discharged. The results, which are tabulated, agree closely with those given by determination with Fe^{2+} and *o*-phenanthroline as indicator. W. J. Wright

2141. **Quinquevalent tungsten as a reducing agent in potentiometric titrations. II. Estimation of ceric salts, bromate and ferricyanide.** A. R. Tourky, I. M. Issa and A. M. Daess (Cairo Univ., Giza, Egypt). *Anal. Chim. Acta*, 1957, **16** (1), 81–86 (in English).—The prep. and properties of standard soln. of W^{5+} have been described previously (*cf. Anal. Abstr.*, 1954, **1**, 927). Difficulties arise in the titration of Ce^{4+} with W^{5+} soln., but the W^{5+} soln. can be titrated with soln. of Ce^{4+} in >7 *N* HCl. Bromate can be titrated with W^{5+} soln. in the presence of H_3PO_4 (up to 2·5 *N*), or the W^{5+} soln. can be titrated with the soln. of BrO_3^- in strong HCl. Ferricyanide is titrated with W^{5+} soln. in 0·5 to 5 *N* H_3PO_4 or 8 to 9 *N* HCl. Separate

inflections for $Fe(CN)_6^{3-}$ and Fe^{3+} are obtained when the two are titrated in the same soln. with W^{5+}. Similarly $Cr_2O_7^{2-}$ and Fe^{3+} can be titrated in the same soln., so also can VO_3^- and Fe^{3+}, but $Cr_2O_7^{2-}$ and $Fe(CN)_6^{3-}$, and $Cr_2O_7^{2-}$ and VO_3^-, respectively, do not yield separate inflections. The soln. of W^{5+} used in these methods range from 0·0035 to 0·011 *N*. W. C. JOHNSON

2142. **The qualitative detection of samarium.** M. N. Ambrozhiĭ and E. F. Luchnikova. *Nauch. Ezhegodnik za* 1954 *g., Saratov Univ.*, 1955, 486–487; *Ref. Zhur., Khim.*, 1956, Abstr. No. 51,126.—Dissolve the sample of rare-earth oxides by warming with the min. quantity of conc. HCl. Cool in ice and pass gaseous HCl through the soln. for 30 min. Separate the crystals of the trichlorides by decantation, dissolve them in abs. ethanol and reduce with magnesium chips while passing gaseous HCl. The red $SmCl_2$ collects on the surface of the Mg (Fe must be absent). Remove the $SmCl_2$ and Mg, treat with CS_2 or formaldehyde soln. and filter off the Mg. To a portion of the filtrate add a few drops of molybdophosphoric acid soln. A green ppt. confirms the presence of Sm. To a second portion of the soln. add an ethanolic soln. of $HgCl_2$; a white ppt. of HgCl also confirms Sm. To a chip of Mg covered with $SmCl_2$ add dil. H_2SO_4 dropwise; the orange $SmSO_4$ is formed, which is unstable in air. C. D. KOPKIN

2143. **Electrophoresis. XVIII. Separation of rare-earth elements.** Masafumi Maki (Japan Women's Univ., Takataminami-cho, Bunkyo-ku, Tokyo). *Japan Analyst*, 1956, **5** (10), 571–573.—The electrophoretic separation (400 V, 3 hr.) of La^{3+}, Ce^{3+}, Pr^{3+}, Nd^{3+} and Y^{3+} was studied with a mixture of citric acid ($\simeq$0·05 *M*) and NaCl (0·05 *M*) at various pH values. At a pH $<$2·8 the ions migrate towards the cathode and at pH $>$3·0 towards the anode. Their separation, except that of Pr^{3+} and Nd^{3+}, is effected at pH 2·8 and 3·0 (velocity of migration, La^{3+} −0·33, Ce^{3+} −0·43, Pr^{3+} and Nd^{3+} −0·33, and Y^{3+} −0·27 μ per sec. per volt per cm at pH 2·8; La^{3+} 0·19, Ce^{3+} 0·30, Pr^{3+} 0·35, Nd^{3+} 0·37, and Y^{3+} 0·41 at pH 3·0). K. SAITO

2144. **Separation of the rare earths by means of the fractional precipitation of their carbonates.** W. Fischer, J. Müller and K. E. Niemann. *Z. anorg. Chem.*, 1956, **282**, 63–79.—Yttrium is by far the major component (usually over 50%) of all naturally occurring yttrium earths. With the usual methods of separation of the rare earths, yttrium occurs at about the mid-point of the lanthanide group, usually in close association with holmium. As a result of this and of the considerable quantities of yttrium present, the element exerts a weighting influence which interferes greatly with fractionation. If, however, the earths are pptd. as the carbonates by the introduction, into a solution of the nitrates, of CO_2 and ammonium hydrogen carbonate at 0°, the yttrium moves out of the normal order. For, while the carbonates of the remaining earths are pptd. in the order of their atomic numbers from Pr, Nd, Sm, La to the elements of the yttrium earths, beginning with Gd and ending with Lu, yttrium carbonate has a greater solubility than the carbonate of any of the lanthanides. L. S. ADLER

2145. **Improved apparatus for the gravimetric determination of carbon dioxide.** E. W. Blank and R. M. Kelley (Colgate-Palmolive Co., Jersey City, New Jersey, U.S.A.). *J. Amer. Oil Chem. Soc.*, 1956, **33** (2), 75–76.—A new apparatus for the determination of CO_2 in soap and synthetic detergents is described and illustrated. A wt. of sample sufficient to yield 0·15 g of CO_2 is decomposed by 20 ml of a mixture of equal vol. of conc. HCl and water. The apparatus is heated to boiling-point over a small flame for 15 to 20 min. while air is slowly drawn through; aspiration is continued after removing the flame, until the flask has cooled to room temp. The CO_2-absorbing tube is removed and weighed. Results obtained are in close agreement with those obtained by the use of the A.O.C.S. official method and it is claimed that the apparatus occupies much less space than the official one. P. HAAS

2146. **Detection of cyanides.** P. W. McConnaughey. U.S. Pat. 2,728,639; date appl. 27.12.55.—The presence of HCN or vinyl cyanide in air or other gases is detected by a reagent consisting of 0·02 g of *o*-tolidine, 0·02 g of $CuSO_4$ (anhyd.) and 2·5 g of glycerol on 10 g of an inert carrier such as silica gel. A blue or blue-green colour is observed with as little as 0·54 μg of HCN per litre of air. The reaction can be made roughly quantitative. N. E.

2147. **New method for the rapid estimation of silica by insolubilisation in glycerol.** C. Liteanu, G. Rusu and C. Strusievici. *Stud. Cercet. Chim., Bucharest*, 1955, **3** (1–2), 55–59.—After evaporating the melt from the alkaline fusion of the sample to dryness with conc. HCl, dehydration of the silica is completed by boiling for 5 min. with glycerol (150° to 170°). Water and a little conc. HCl are added, and the silica is filtered off, calcined and treated with HF. The whole analysis requires $\simeq$2·5 hr. The results are in good agreement with those given by the methods employing gelatin or $HClO_4$, and the precision is greater. J. H. WATON

2148. **Rapid colorimetric determination of silica in silicates.** Toshikazu Tarutani (Dept. of Anal. Chem., Tokyo Inst. Tech., Ookayama, Tokyo). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1956, **77** (9), 1324–1327.—A simple procedure for use in rock analysis is suggested. The time is minimised by preparing solutions direct from the fused melt; volatilisation of Si as SiF_4, in the presence of F in rocks, is thus avoided. To prevent coagulation of SiO_2, the basic soln. should be diluted to give a concn. of $<$0·1 mg of SiO_2 per ml, before acidification. Sodium fluoride (masking agent for Fe) must be added after the coloration of the molybdosilicate has reached its max., and the extinction must be measured rapidly after the addition. *Procedure*—The sample (0·1 g) is mixed with Na_2CO_3 (0·2 g) and NaOH (2 g), fused for 0·5 to 1 hr. and the melt is leached with water. When no ppt. remains, the soln. is made up to 250 ml and a 5-ml portion is diluted to $\simeq$40 ml, neutralised, made up to 50 ml, treated with 3 *N* H_2SO_4 (2 ml) and 10% $(NH_4)_2MoO_4$ (2 ml), and the extinction is measured. When a ppt. is formed in the leaching of the melt, it is filtered off, dissolved in 18 *N* H_2SO_4 (8 ml) and the soln. is made up to 250 ml; a 10-ml portion is diluted to 50 ml and treated with $(NH_4)_2MoO_4$ soln. and 2% NaF soln. (4 ml); the filtrate is treated as described above, and the total SiO_2 determined. K. SAITO

2149. **New method for the rapid estimation of silica in silicates (clay, kaolin and bauxite).** C. Liteanu, C. Strusievici and G. Rusu. *Stud. Cercet. Chim., Bucharest*, 1955, **3** (1–2), 61–66.—The siliceous material is decomposed by fusion with $KHSO_4$, the heating being continued for 3 to 4 min. at 800° until the mass solidifies. The melt is extracted with HCl (1:2) or with H_2SO_4 (1:3), after which the soln. is boiled and filtered. The residue is calcined and treated with HF. The analysis requires a max. of 2 hr. The method is as rapid and as accurate as those employing gelatin or $HClO_4$

J. H. WATON

2150. **Flame-colour test for tin.** Zenzo Tamura and Kazuko Kawahara (Pharm. Inst., Med. Fac., Tokyo Univ., Hongo, Tokyo). *Japan Analyst*, 1956, **5** (10), 559–561.—The mechanism of Meissner's flame-colour test (*Z. anal. Chem.*, 1930, **80**, 247) was studied under various conditions, and the reaction appears to be characteristic of $SnCl_4$, and not, as Meissner suggested, of SnH_4. Bivalent Sn in HCl is easily oxidised to Sn^{4+} in the flame and gives a blue tint to the flame; Sn^{4+} are reduced to metallic Sn when the soln. is evaporated to dryness (*e.g.*, on the surface of a test-tube inserted into the flame). K. SAITO

2151. **Turbidimetric determination of trace tin in refined copper.** C. M. Dozinel and H. Gill (Lünen, Westfalen, Germany). *Chemist Analyst*, 1956, **45** (4), 105, 109.—The rapid turbidimetric procedure described in which 4-hydroxy-3-nitrobenzenearsonic acid is used as reagent has been applied successfully to refined copper containing 0·0001 to 0·0050% of tin. O. M. WHITTON

2152. **A rapid oxidimetric method for the estimation of lead dioxide.** J. Bitskei (Anorg.-chem. Inst., Tech. Univ., Budapest). *Z. anal. Chem.*, 1957, **154** (4), 259–262.—By using methyl red or indigo carmine as indicator, hydrazine can be titrated in HCl soln. with 0·1 *N* NaOCl according to the equation—

$$2NaOCl + H_2N.NH_2 = 2NaCl + 2H_2O + N_2$$

(*cf.* Bitskei and Petrich, *Mag. Kém. Lapja*, 1947, **14**, 231). This reaction can be applied to the estimation of any material that destroys hydrazine in an oxidation - reduction process, *e.g.*, lead dioxide. The sample is heated with excess of hydrazine sulphate soln. in HCl in the presence of $MnCl_2$, when it reacts according to the equation—

$$2PbO_2 + H_2N.NH_2 + 4H^+ = 2Pb^{2+} + 4H_2O + N_2.$$

The excess is titrated with NaOCl. G. BURGER

2153. **Determination of oxygen in titanium and titanium alloys.** M. Codell and G. Norwitz. U.S. Pat. 2,731,330; date appl. 17.1.56. See *Anal. Abstr.*, 1956, **3**, 94.

2154. **Direct spectrophotometric determination of zirconium with SPADNS.** Gurupada Banerjee (Ass. Cultivation of Science, Calcutta, India). *Anal. Chim. Acta*, 1957, **16** (1), 62–66 (in English).—The method depends on the formation of a crimson - pink complex of Zr with SPADNS (*cf. Anal. Abstr.*, 1957, **4**, 2156). *Procedure*—To an aliquot (≯ 4 ml) of the sample soln. containing up to 56 μg of ZrO_2 add 3 ml of a 0·1% soln. of SPADNS and make up the volume to 25 ml with 0·5 *N* HCl. Measure the extinction at 580 mμ, against a reagent blank, in a Unicam SP600 spectrophotometer, with a 1-cm cell. W. C. JOHNSON

2155. **Thermolysis of zirconium precipitates. I. Salts of organic acids.** W. W. Wendlandt (Texas Technol. Coll., Lubbock, U.S.A.). *Anal. Chim. Acta*, 1957, **16** (2), 129–134 (in English).—Thermogravimetric studies of Zr phthalate, phenoxyacetate, cinnamate, diphenate, 8-quinoline complex, *p*-bromomandelate, benzilate, *m*-tolyloxyacetate, salicylate and benzoate are reported. The pptd. salts are stable in varying degree, the first losses of wt. occurring between 45° and 160°, and the final const. wt. (ZrO_2) between 500° and 790°. The bromomandelate has excellent thermal stability up to 150°, but Zr diphenate and benzilate are stable only up to 50° and 85°, respectively. The Zr - 8-quinoline chelate corresponds very closely to $2ZrO_2.3C_9H_7ON$; its decomposition, starting at 45°, becomes rapid at 200°, with complete formation of ZrO_2 at 610°. The conditions of pptn. and the use of organic acids, *e.g.*, phthalic, cinnamic, diphenic, benzilic, salicylic or benzoic, to separate Zr from other metals, *e.g.*, Hf, U, and cerite rare-earths, are discussed briefly. W. J. BAKER

2156. **Direct spectrophotometric determination of thorium with SPADNS.** Gurupada Banerjee (Ass. Cultivation of Science, Calcutta, India). *Anal. Chim. Acta*, 1957, **16** (1), 56–61 (in English).—The blue - violet complex of Th and SPADNS [Na 2-(*p*-sulphophenylazo)-1; 8-dihydroxynaphthalene-3; 6-disulphonate] is used for the determination of quantities of Th down to 0·04 μg in 25 ml of final vol. *Procedure*—To an aliquot of the soln., containing up to 5 μg of Th, add sufficient 0·01 *N* HCl to produce a pH of 2·6 to 3·6 and 3 ml of a 0·1% soln. of SPADNS; dilute with water to 25 ml and measure the extinction at 580 mμ, against a reagent blank, in a Unicam SP600 spectrophotometer, with a 1-cm cell. Moderate amounts of U and of rare earths cause no appreciable interference, but Zr, F^-, PO_4^{3-}, oxalate and large amounts of SO_4^{2-} must be absent. W. C. JOHNSON

2157. **Determination of metal ions by means of their oxalates. V. Determination of thorium.** Kyozo Kawagaki (Fac. of Engng, Niigata Univ., Nagaoka). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1956, **77** (10), 1459–1461.—Experimental conditions for the quant. pptn. of Th with oxalate were studied. The amount of oxalic acid, the best precipitant, must be greater than twice the equiv. of the Th, and the ppt. must be set aside for >30 min. before being filtered off. The ppt. is washed with water, dissolved in dil. H_2SO_4, and titrated with $KMnO_4$. Satisfactory results are also obtained by the use of a known amount of oxalic acid, followed by titration of the excess. K. SAITO

2158. **Rhodanine-N-salicylic acid and its use in the estimation of thorium.** M. K. Rout (Mayurbhanj Chem. Lab., Ravenshaw College, Cuttack, India). *J. Indian Chem. Soc.*, 1956, **33** (9), 683–684.—A method of estimating Th based on the complex it forms with rhodanine-N-salicylic acid is given. A structure is proposed for the complex which is supported by its ignition to ThO_2. *Procedure*—Thorium nitrate soln. (0·2 to 0·8%) (25 ml) is made neutral to Congo red with aq. NH_3. An excess of a 1% soln. of the reagent is added (fourfold excess is adequate). The soln. is warmed on a water bath for 15 min. and then set aside overnight. The ppt. is collected in a G3 crucible, washed with hot water, and dried at 110° to 115°. The complex is weighed and the percentage of Th calculated from the

proposed structure. The prep. of the reagent is described. C. A. SLATER

2159. **Thorium complex of 1:8-dihydroxy-2:7-dinitrosonaphthalene-3:6-disulphonic acid [dinitrosochromotropic acid]. III. Complexiметric determination of thorium with EDTA.** S. K. Datta (Gov. Coll., Darjeeling, India). *Anal. Chim. Acta*, 1957, **16** (2), 115–120 (in English).—Dinitrosochromotropic acid (0·025% aq. soln.) can be used as indicator in the titration of thorium with 0·012 *M* EDTA (disodium salt) because its pinkish-violet complex (formed initially) with Th is less stable than the colourless Th - EDTA complex, and decomposes into the free acid (red) when most of the Th has reacted with EDTA. The end-point is very sharp especially in the reverse direction. Concn. as low as 3 mg of Th per 50 ml of soln. can be determined accurately at pH 2·2 to 3·5. Iron can be masked with ascorbic acid, but Cu, Pb, Sn, U, Au, Ti and rare-earth elements must be removed by pptn. of Th with *o*-anisilic acid at pH 3·6 and 90°. Phosphate, citrate, tartrate and F^- interfere. The accuracy is as high as for the same titration with Xylenol orange indicator. W. J. BAKER

2160. **Spectrophotometric determination of silicon in thorium oxide.** O. Menis and D. L. Manning (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chim. Acta*, 1956, **16** (1), 67–75 (in English).—*Procedure*—Dissolve 0·5 to 1 g of the thorium oxide by stirring and heating to 80° with 5 ml of conc. HNO_3, 25 ml of water and 1 to 2 drops of conc. HF (48%) in a platinum dish. Cool, mix with 30 ml of a saturated soln. of H_3BO_3 in a polyethylene cylinder and dilute with water to 100 ml. Take an aliquot containing 2 to 40 μg of Si, dilute with water to ≃50 ml in a polyethylene beaker, add 15 ml of 10% (w/v) ammonium molybdate soln. and aq. NH_3 (SiO_2-free) to produce a pH between 0·9 and 1·4. Set aside for 5 min., then add 20 ml of 20% (w/v) tartaric acid soln. and, after a further 2 min., 1 ml of a solution prepared by dissolving 7 g of Na_2SO_3 in 100 ml of water, adding 1·5 g of 1-amino-2-naphthol-4-sulphonic acid and a soln. of 90 g of $NaHSO_3$ in 800 ml of water and diluting with water to 1 litre. Adjust the pH to 3·0 ± 0·1, stir to dissolve the pptd. thorium molybdate, and dilute to 100 ml. Measure the extinction at 840 mμ in a 5-cm cell and deduct the value obtained for a reagent blank. Prepare a calibration curve with known amounts of Si. The coeff. of variation under the conditions for the prep. of the standard curve is 5% and for samples it is 13%. Phosphate (>400 μg) does not interfere, but large quantities of Fe and electrolytes must be absent. W. C. JOHNSON

2161. **Determination of nitrates in aqueous solution.** J. Ungar (The Permutit Co. Ltd., Gunnersbury Ave., London). *J. Appl. Chem.*, 1956, **6** (6), 245–247.—Indigo carmine is quant. oxidised and decolorised by NO_3^- to isatinsulphonic acid. The sample is acidified with conc. H_2SO_4 and titrated with acidified indigo carmine soln. until the colour just persists when the soln. is boiled. The accuracy is within ±2% and results are superior to those by the phenoldisulphonic acid method. Interfering ions are Cr^{3+}, Mn^{2+}, Ca^{2+}, Cd^{2+} and Fe^{3+}, but these can be removed by Zeo-Karb 225. Nitrite interferes seriously, and should be oxidised with bromine to NO_3^-; NO_2^- are determined on a separate aliquot. F. J. M.

2162. **Photometric determination of phosphorus as molybdovanadophosphoric acid.** O. B. Michelsen (Defence Res. Estab., Kjeller, Norway). *Anal. Chem.*, 1957, **29** (1), 60–62.—A modified normal procedure, in which very dil. reagents are used, enables concn. as low as 0·1 μg of P per ml (*e.g.*, of H_3PO_4 soln.) to be determined spectrophotometrically at 315 mμ. Beer's law is valid and the absorption background is reduced; a final acidity of 0·02 to 0·07 *N* (the latter value is preferred) ensures max. extinction within 2 min. The accuracy is within ±1% at concn. of P >1 μg per ml, and is within ±3% at lower concn. W. J. BAKER

2163. **New reagent for the colorimetric and spectrophotometric determination of phosphorus, arsenic and germanium.** L. Prat Perez (Univ. of Salamanca, Spain). *Acta Salmanticensia, Serie de Ciencias*, 1956, **2** (2), 66 pp.—The reagent consists of a mixture of ammonium molybdate and $MoCl_5$; the optimum ratio of Mo^{6+} to Mo^{5+} is 3:2, the stability increasing with acidity. The concn. proposed are Mo^{6+} (0·12 *M*), Mo^{5+} (0·08 *M*), H_2SO_4 (10 *N*) and HCl (3 *N*), 1·5 ml of reagent being employed. In the presence of P the blue colour formed shows an absorption max. at 840 mμ and Beer's law is obeyed up to 160 μg of P_2O_5 (in 50 ml). The composition of the blue complex is independent of the Mo^{6+} to Mo^{5+} ratio, the quantity of P and the temp., but the sensitivity increases with temp., and 15 min. heating on a water bath is proposed. Interference by pptn. is caused by Ba^{2+}, Sb^{3+}, Bi^{3+} and Pb^{2+}, and AsO_4^{3-}, GeO_3^{2-} and WO_4^{2-} give the same reaction; Fe^{3+}, ClO_4^- and Ca^{2+} do not interfere; Fe^{3+} and NO_3^- interfere at >20 p.p.m., and $C_2O_4^{2-}$ at >100 p.p.m. The reagent may be used for determining P in soils, by using an extract of pH 3·2, and reducing Fe^{3+} with sulphite. Arsenic and Ge may be similarly determined, the absorption max. being at 850 mμ and 830 mμ, respectively, and Beer's law being obeyed up to 345 μg of As_2O_3 and 220 μg of GeO_2. The reagent is unsuitable for the determination of SiO_2.
L. A. O'NEILL

2164. **Analytical chemistry of the acids of quinquevalent phosphorus. V. Determination of pyrophosphoric acid—the barium procedure.** J. Fischer and G. Kraft (Inst. f. anorg. Chem., Goethe Univ., Frankfurt/Main). *Z. anal. Chem.*, 1957, **154** (4), 245–253.—Pptn. by $Ba(OH)_2$ with determination of the excess has previously been used. This offers no fundamental advantage over pptn. with Ca^{2+} and Zn^{2+}. It is now shown that pyrophosphate can be efficiently estimated by pptn. with a neutral salt of Ba and titration of the H^+ produced. *Procedure*—Neutralise the pyrophosphate (0·1 *M*) to the secondary salt (pH 4) and precipitate with a 10% soln. of $BaCl_2$. Pptn. is complete in 3 min. Titrate the HCl formed with 0·1 *N* NaOH (glass electrode). During the titration any CO_2 is driven off with a stream of CO_2-free gas. The reproducibility is within ±1%. The composition of the ppt. and the effect of change of conditions are discussed. Orthophosphate, metaphosphate and higher condensed phosphates do not interfere. Phosphates of medium chain length (tri- and tetra-phosphates) are co-pptd. and give a false result. Graham's salt, if present, must be pptd. with $BaCl_2$ at pH 2 and filtered off. G. BURGER

2165. **Determination of arsenic in iron, steel, ores and sintered ores.** Arihiro Tominaga and Shiro Watanabe (Kamaishi Iron Mfg. Works, Suzuko-cho,

Kamaishi, Iwate Prefecture). *Japan Analyst*, 1956, **5** (9), 495–499.—Oxidative fusion followed by iodimetric titration was employed for the determination of As (0·1 to 5%) in limonite and sintered ores. Arsenate quantitatively oxidises KI in a strongly acid soln. (>16% HCl or >33% H_2SO_4). When the fused cake is leached, As (≃0·1% of its total) is adsorbed by the $Fe(OH)_3$, but this is negligible in a rapid analysis. *Procedure*—The sample (1 to 2 g) is fused with a mixture (7 g) of Na_2O_2 and Na_2CO_3 (2 + 1) for 15 min., leached with hot water (100 ml) and made up to 250 ml. A 50-ml portion is neutralised to phenolphthalein with H_2SO_4 (1 + 1), made acid with a further 10 ml of the H_2SO_4 and boiled for 5 min. The product is cooled, treated with conc. HCl (20 ml) and KI (1 g) and titrated with $N/30$ $Na_2S_2O_3$ after 15 min. For a colorimetric determination, a small amount of As (<0·05%) in steel or ore is co-pptd. with MnO_2, dissolved in H_2SO_4 containing H_2O_2 and treated with $(NH_4)_2MoO_4$. The yellow soln. is shaken with amyl alcohol to remove molybdophosphate, and the aq. layer, which retains all the molybdoarsenate, is treated with hydrazine sulphate before measuring the extinction at 700 mμ. The working curve is linear for <0·20 mg per 100 ml. *Procedure*—Steel (0·5 g) is dissolved in HNO_3 (1 + 1, 18 ml) and boiled with 5% $MnSO_4$ (7 ml) and water (100 ml); $KMnO_4$ (3%, 3 ml) is gradually added and the ppt. is filtered off. The MnO_2 is dissolved in 3 *N* H_2SO_4 and H_2O_2, boiled, neutralised with 3 *N* NaOH and mixed with 1% $(NH_4)_2MoO_4$ (7 ml) and H_2SO_4 to produce 50 ml of 0·2 to 0·3 *N* H_2SO_4 soln. The product is shaken with 5-ml portions of amyl alcohol until the organic layer becomes colourless. The acidity of the aq. layer is increased to 0·4 *N* and made up to 100 ml with 1% hydrazine sulphate (10 ml) and water, and the extinction is measured. Ores (0·5 g) are fused with the mixture (3 g) of Na_2O_2 and Na_2CO_3 (2 + 1) for 5 min., leached with water, dissolved by addition of HNO_3 (1 + 1) and H_2O_2, neutralised and again made acid for similar treatment. K. SAITO

2166. **Use of trihydroxyfluorone derivatives in colorimetric analysis. Determination of antimony.** V. A. Nazarenko and N. V. Lebedeva. *Zhur. Anal. Khim.*, 1956, **11** (5), 560–565.—The use of 2:3:7-trihydroxy-9-fluorone for colorimetric determination of Sb (*cf. Anal. Abstr.*, 1956, **3**, 1294) is studied in greater detail. The max. absorption of the complex occurs at 530 mμ, the molar extinction coeff. is 34,160, and p*K* for the equilibrium is 6·88. Soln. stabilised by gelatin follow Beer's law. Determinations can be carried out satisfactorily over the concn. range 0·05 to 6 μg per ml. G. S. SMITH

2167. **The oxinates of antimony. Chemical determination of antimony by means of oxine.** Ichiro Kudo (Inst. Polytech., Osaka City Univ., Minamiogi-machi, Kita-ku, Osaka). *Japan Analyst*, 1956, **5** (10), 556–559.—Tervalent Sb yields the same oxinate, $Sb(C_9H_6ON)_3$, in neutral soln., in the presence or absence of tartrate. This was confirmed by chemical analysis and X-ray crystallography, but is contrary to Pirtea's finding (*Z. anal. Chem.*, 1939, **118**, 26) that an antimonyl compound is formed in the presence of tartrate. Quinquevalent Sb yields a yellow ppt. with oxine in HCl of concn. >10 *N*, which was identified as $Sb(C_9H_6ON)_2Cl_3$. A ppt. of similar composition is formed with 5:7-dichloro-oxine or 5:7-dibromo-oxine. This reaction is characteristic of Sb^V and can be used for its separation from most ions, including those of As and Bi. The ppt. is washed with conc. HCl, saturated with oxine, then with conc. HCl, and kept in a desiccator containing NaOH until the weight is constant. The ppt. are readily decomposed when heated. K. SAITO

2168. **Determination of antimony in indium antimonide.** M. C. Bachelder and P. M. Sparrow (Inst. Study of Metals, Univ., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 149–150.—Approx. 200 mg of Sb in In_2O_3 or InSb (pure or admixed with Sb) can be determined to within 0·2% by fusing the sample with six times its wt. of a mixture (1 + 1) of anhyd. Na_2CO_3 and pure S in a covered crucible, dissolving the melt in the min. of 12 *N* HCl, oxidising the pptd. S with $KClO_3$, and then titrating the Sb iodimetrically in the Cl-free soln. to the potentiometric end-point. Solid KCl is added to the dissolved melt to form the less volatile $SbCl_4^-$. A blank should be run on the reagents and acid. There is no loss of Sb. W. J. BAKER

2169. **Bismuthiol II as an analytical reagent. I. Estimation of bismuth.** A. K. Majumdar and M. M. Chakrabartty (Jadavpur Univ., Calcutta). *Z. anal. Chem.*, 1957, **154** (4), 262–267 (in English).—Bismuthiol II (5-mercapto-3-phenyl-2-thio-1:3:4-thiadiazol-2-one) forms coloured ppt. with several ions and is suitable for the detection of small quantities of Bi (identification limit, 1·2 μg; sensitivity, 1 in 2·8 × 10^4). A study has been made of the effect of acidity, reagent concn., and interfering ions. Bismuth is separated from certain ions by pptn. from acid soln., from others by pptn. from acid soln. in the presence of citrate or tartrate, and from a third group by pptn. of the unwanted ions by means of the reagent from neutral soln. in the presence of citrate or tartrate and estimation of Bi in the acidified filtrate. Oxidising ions and ions that form insol. compounds with Bi or with the reagent interfere. G. BURGER

2170. **New accurate and rapid gravimetric method for the estimation of bismuth.** G. Spacu and F. Popea (Inorg. and Anal. Chem. Lab., "C.I. Parhon" Univ., Bucharest, Romania). *Stud. Cercet. Chim., Bucharest*, 1955, **3** (3–4), 175–181.—Bismuth is pptd. quant. as the complex $[Coen_2Cl_2]_3[BiCl_6]$ (en = ethylenediamine) by the addition of $[Coen_2Cl_2]Cl$ (*trans* isomer) in the presence of ethanol and NH_4Cl. The soln., which should be feebly acid with HCl, must be cooled to between 0° and 5°. After filtration, the ppt. is washed with ethanol and with ether, and is dried in a vacuum desiccator. The ions Na^+ K^+, NH_4^+, Ca^{2+}, Cl^-, acetate and SO_4^{2-} do not interfere. The analysis can be performed in 90 min. J. H. WATON

2171. **Determination of bismuth in metallurgical products: Addendum.** J. Kinnunen and B. Wennerstrand (Outokumpu Oy, Metalworks, Pori, Finland). *Chemist Analyst*, 1956, **45** (4), 109.—The colour of the Bi - diethyldithiocarbamate complex in the photometric procedure for Bi (*Anal. Abstr.*, 1955, **2**, 1194) has been shown to be unstable to light in $CHCl_3$, CCl_4, and in amyl and ethyl acetates, so that a dark room and immediate measurement are required with these solvents. Alternatively, *n*-pentanol and *n*-butanol can be used in the published procedure as the colour of the bismuth complex is stable in these solvents. O. M. WHITTON

2172. **Semi-micro colorimetric determination of vanadium in minerals and ores.** O. M. Silina. *Sb. Nauch. Tekhn. Inform. Ministerstvo Geol. i Okhranÿ Nedr*, 1955, (1), 127–128; *Ref. Zhur., Khim.*, 1956, Abstr. No. 54,766.—The formation of the tungstovanadophosphate complex in H_2SO_4 soln. is used. With a sample wt. of 0·1 to 0·3 g, materials having a high content of SiO_2 can be analysed for contents of V between 0·01 and 3%. The error is ±0·005% of the sample wt. at very low contents, and ±0·1% at 1% of V. Interference by Cr occurs only when its content greatly exceeds that of V.

G. S. SMITH

2173. **Colorimetric determination of niobium and tantalum in ores.** F. V. Zaĭkovskiĭ (All-Union Sci. Res. Inst. of Mineral Raw Materials). *Zhur. Anal. Khim.*, 1956, **11** (5), 553–559.—By extraction under suitable conditions of the catechol complexes, Nb, Ta and Ti can be separated from each other and from Fe, Zr, W, Al and other elements (*cf. Anal. Abstr.*, 1957, **4**, 1184). With ores containing only traces of Ti, the sample (0·1 to 1 g) is treated with 5 ml of HF and then with 10 ml of dil. H_2SO_4 soln. (1 + 1), and the ignited residue is fused with $K_2S_2O_7$. The melt is dissolved in 25 ml of 3% ammonium oxalate soln. and the insoluble matter is fused with $K_2S_2O_7$, dissolved in 10 to 15 ml of 3% ammonium oxalate and added to the main soln. With ores containing more than 5 to 6 mg in the sample wt., the residue after treatment with HF and H_2SO_4 is heated with 15 ml of conc. HCl, the soln. is evaporated to half its bulk and then boiled for 30 min. with 300 ml of 1% tannin in dil. HCl (1 + 9) in the presence of filter-paper pulp. The ppt. is collected on the following day, then ignited and fused with $K_2S_2O_7$, etc., as described above. The soln. is mixed with catechol to give a concn. of 6% and partially neutralised with aq. NH_3. Saturated Na_2SO_3 is added until the soln. darkens (reaction with Fe) followed by a 5-ml excess (pH 7 to 8) and the soln. in a separating funnel is extracted with 12 to 15 ml of *n*-butanol that has been saturated with water. The extraction is repeated with a further 8 to 10 ml of *n*-butanol, the combined extracts are evaporated nearly to dryness, the residue is dissolved in 20 to 30 ml of 10% H_2SO_4 and the cold soln. is mixed with 8 to 10 ml of 3% cupferron soln. The ppt. is collected after 2 to 3 min. and ignited. The residue containing Nb and Ta together with some of the Ti is fused with 0·3 to 0·4 g of $K_2S_2O_7$, the melt is dissolved in 22 to 25 ml of 3% ammonium oxalate soln., the pH is adjusted to between 2·5 and 3 (Congo red) by means of Na_2SO_3 and dil. H_2SO_4, 4 g of catechol is added and the soln. is extracted with 8 to 10 ml of *n*-butanol. After four or five extractions the aq. soln. is free from Ti and Ta. The aq. layer contains all the Nb. The first three extracts are combined and extracted two or three times with 10 to 15-ml portions of 3% H_2SO_4. The solvent phase containing the Ta is evaporated to dryness, the residue is mixed with 20 ml of water and evaporated nearly to dryness, this residue is dissolved in 10 to 20 ml of 10% H_2SO_4 and, after addition of 1 ml of a 1% soln. of a salt of Zr, 8 to 10 ml of cupferron soln. is added. The ppt. is collected and fused with 0·2 to 0·3 g of $K_2S_2O_7$, the melt is dissolved in 20 ml of 3% ammonium oxalate soln., 1 g of catechol is added, the pH is brought to 7 to 8 (litmus) and the Ta is extracted with 8 to 10 ml of *n*-butanol. The extract is evaporated with H_2SO_4 and then with water, and the residue is dissolved in 10 to 20 ml of 10% H_2SO_4. After addition of 1 ml of a 1% soln. of a salt of Zr, the soln. is treated with 8 to 10 ml of cupferron soln., the ppt. is collected, ignited and fused with $K_2S_2O_7$, and the melt is dissolved in 5 to 10 ml of 3% ammonium oxalate soln. An aliquot portion (1 to 3 ml) is mixed with 0·5 ml of 5% H_2SO_4 soln. and 1 ml of 20% catechol soln. containing $SnCl_2$ [50 ml of 20% catechol soln. is treated with 2 ml of a 15% soln. of $SnCl_2$ in dil. HCl (1 + 9)] and the colour is compared with standards to give the content of Ta. The aq. layer containing the Nb is diluted to 25 or 50 ml in a calibrated flask with 3% ammonium oxalate and 0·5 to 5 ml is mixed with 2 ml of the catechol soln., 1 to 3 ml of saturated Na_2SO_3 soln., and sufficient 3% ammonium oxalate soln. to give 10 ml. The extinction is measured after 5 to 6 min., with a blue filter, and the content read from a calibration curve or the colour is compared with standards.

G. S. SMITH

2174. **Determination of sulphur dioxide in gas mixtures.** C. M. Cherrier. U.S. Pat. 2,736,813; date appl. 28.2.56.—The gas being analysed is compared with a sample from which SO_2 has been removed. The absorption of u.v. light passing through the two gases simultaneously is measured by two photo-electric cells; the difference is the measure of the SO_2.

N.E.

2175. **Titration of sulphate following separation with alumina.** J. S. Fritz, S. S. Yamamura and M. J. Richard (Iowa State Coll., Ames, U.S.A.). *Anal. Chem.*, 1957, **29** (1), 158–161.—Milligram amounts of SO_4^{2-} can be separated from most metal ions and large excesses of Cl^-, NO_3^- or ClO_4^- by passing the soln. (adjusted to pH 0·5 to 1 with HCl or $HClO_4$) slowly through a 6-cm column of 80 to 200-mesh activated Al_2O_3. The retained SO_4^{2-} are then eluted with aq. NH_3 (*M* followed by 0·1 *M*), the eluate is passed through a small cation-exchange column (3 in. × 1 in., H form), and an aliquot of the final eluate (100 ml) is titrated with 0·01 *M* $Ba(ClO_4)_2$ in the presence of ethanol and with thorin as indicator. The method is inapplicable when the metal ion forms a slowly reacting sulphate complex. Modified procedures are given for the separation of SO_4^{2-} from Zr^{4+}, Cr^{3+} or Th^{4+} [by complexing these metals with N-hydroxyethylethylenediaminetriacetic acid (Entol)] and for the titrimetric determination of S in organic compounds after fusion with Na_2O_2 in a bomb. Results for 24 widely different organic compounds of S indicate an error $\gtrsim 1\%$. The lower limit for the quant. recovery of SO_4^{2-} on the Al_2O_3 column (chloride or perchlorate form) is 0·5 p.p.m.

W. J. BAKER

2176. **Direct potentiometric titration of the sulphate ion with the indicator electrode Mo | MoO_3 | $BaSO_4$. I.** A. D. Miller (Lensovet Leningrad Technol. Inst.). *Zhur. Anal. Khim.*, 1956, **11** (5), 615–620.—The indicator electrode is prepared from a molybdenum wire 1 mm in diam. with two or three twists at the end. An oxide layer is produced as follows. The wire is repeatedly heated and immersed in molten sulphur, then alternately heated (without immersion in S) and dipped into a saturated soln. of $BaCl_2$ or $Ba(OH)_2$ and finally immersed in 0·2 *N* H_2SO_4. It is preserved in 0·001 *N* Na_2SO_4. The electrode can be used in the direct potentiometric titration of SO_4^{2-} with $BaCl_2$ in neutral or acid soln.

G. S. SMITH

2177. **A colorimetric method for the determination of thiosulphate.** B. Sörbo. (Fac. des Sci., Paris, France). *Biochim. Biophys. Acta*, 1957, **23** (2), 412–416.—The method described is based on the conversion of thiosulphate into thiocyanate ions in the presence of CN^- and Cu^{2+}, followed by the determination of thiocyanate colorimetrically with Fe^{3+}. From 0·05 to 1·5 μ-equiv. of thiosulphate may be determined. Interference by thiocyanate, sulphides or polythionates in the sample may be avoided by the procedures given, and the effects of other ions are discussed. *Procedure*—To 4·2 ml of sample (if buffered, the sample is made alkaline to thymolphthalein added as internal indicator) add 0·50 ml of aq. KCN (0·1 *M*), mix and add 0·30 ml of aq. $CuCl_2$ soln. (0·1 *M*). Mix immediately and add 0·5 ml of Fe^{3+} reagent [100 g of $Fe(NO_3)_3.9H_2O$ and 200 ml of 65% HNO_3 diluted with water to 1 litre]. Measure the extinction at 460 mμ in a 1-cm cell and deduct a "blank" value obtained by adding the Fe^{3+} reagent before the KCN and $CuCl_2$ reagents.
W. H. C. Shaw

2178. **Polarographic determination of selenium.** V. F. Toropova (V.I. Ul'yanov-Lenin Kazan State Univ.). *Zhur. Anal. Khim.*, 1956, **11** (5), 599–601.—The method is based on the reduction of the selenosulphate ion $SeSO_3^{2-}$ at the dropping-mercury electrode. With 0·005 *M* Na_2SeSO_3 in 0·3 *M* $NaNO_3$, $E_{\frac{1}{2}}$ is −1·06 V *vs.* the S.C.E., and is independent of the concn. of NaOH if the pH $\not<10$. To determine Se in Na_2SeO_3, the soln. acidified with HCl is treated with excess of thiourea to precipitate Se, and the soln. containing the ppt. is neutralised with Na_2CO_3 and treated with Na_2SO_3 to dissolve the ppt. The soln. is then diluted to 50 ml and the polarogram obtained.
G. S. Smith

2179. **Separation and determination of microgram amounts of molybdenum.** G. R. Waterbury and C. E. Bricker (Univ. of Calif., Los Alamos, N.M., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 129–135.—Microgram amounts of Mo in plutonium alloys and other samples can be separated and determined by extraction from a 6 *M* HCl - 0·4 *M* HF soln. into *iso*butyl methyl ketone (Hexone), followed by back-extraction of the Mo plus Fe into H_2O. After removal of Fe by pptn. with 0·2 *M* NaOH from the aq. extract (freed from F^-), the Mo is determined spectrophotometrically at 350 mμ, by using its coloured complex with chloroanilic acid (0·1%) in 1·4 *M* $HClO_4$ at 20°. After correcting for the extinction of the blank, the concn. of Mo is calculated from a calibration curve. The mean recovery of Mo is 99·8% with a standard deviation of 1·6% (32 analyses, 19 to 96 μg of Mo); reproducibility is within 3% for samples containing > 15 μg. Tungsten, Sn and Bi interfere seriously even in very small amounts; other interfering ions and variables affecting the result are discussed. A single analysis takes $\simeq$6 to 7 hr. but ten analyses can be made simultaneously in $\simeq$2 days.
W. J. Baker

2180. **Polarographic catalytic current of molybdenum and its use for determining microgram amounts of molybdenum.** S. I. Sinyakova and M. I. Glinkina (Vernadskiĭ Inst. of Geochem. and Anal. Chem., Acad. Sci., USSR, Moscow). *Zhur. Anal. Khim.*, 1956, **11** (5), 544–552.—In *M* $NaClO_4$ - 0·75 *M* H_2SO_4 the polarographic limiting current of Mo at the dropping-mercury electrode is determined by the rate of the chemical reaction as well as by diffusion. By means of the catalytic wave, Mo in concn. as low as 10^{-6} *M* can be determined with an error of $\pm$10%. To determine Mo in minerals, etc., the sample (1 to 2 g) is dissolved in 40 ml of aqua regia and the soln. is evaporated to 5 ml. It is then evaporated to fuming with 2 ml of H_2SO_4, the residue is boiled with water, the filtered soln. is evaporated to 10 ml and treated with dry Na_2CO_3 until a ppt. appears, and one or two drops of dil. H_2SO_4 soln. (1 + 1) are added to dissolve the ppt. The soln. in a separating funnel is treated with H_2SO_4 to give an acid concn. of 1 to 2%, then shaken with 2 ml of 2% ethanolic benzoin α-oxime soln., followed by shaking with 5 ml of $CHCl_3$. The solvent layer is removed and the aq. layer re-extracted twice with 5 ml of $CHCl_3$. The combined extracts are evaporated to dryness and the residue is heated with a mixture of 2 ml of conc. H_2SO_4, 5 ml of conc. HNO_3 and five to ten drops of $HClO_4$ until fuming ceases. The hot residue is treated with 5 ml of a mixture *M* in $NaClO_4$ and 0·75 *M* in H_2SO_4, hydrogen is passed through the soln. for 5 min. and the polarogram between 0 and −0·6 V *vs.* the S.C.E. is recorded. The content of Mo is found by means of a calibration curve. No interference is caused by Fe, Cu, Pb, Zn, V and W, and with contents $\simeq 10^{-4}$ % the error in determining Mo is $\pm$6%.
G. S. Smith

2181. **Micro-heterometric determination of molybdic acid with benzoin α-oxime.** M. Bobtelsky and I. Julius (Hebrew Univ., Jerusalem, Israel). *Anal. Chim. Acta*, 1957, **16** (1), 75–80 (in English).—Soln. of MoO_4^{2-}, containing $\simeq$1 mg of Mo and 1 to 3 ml of *M* HCl or HNO_3 in 20 ml, are titrated with alcoholic 0·01 *M* benzoin α-oxime by the heterometric method (*cf. Anal. Abstr.*, 1956, **3**, 12). The titration can be conducted in the presence of the ions of most of the common metals, as well as WO_4^{2-} and VO_3^-. Errors up to 3% are recorded.
W. C. Johnson

2182. **Improvements in the accuracy of methods of determining tungsten in sulphide ores.** V. L. Khukhiya and T. G. Matsaberidze. *Sb. Nauch. Tekhn. Inform. Ministerstvo Geol. i Okhrany Nedr*, 1955, (1), 126–127; *Ref. Zhur., Khim.*, 1956, Abstr. No. 54,768.—Sulphide ores with high contents of W are fused with $K_2S_2O_7$, the aq. extract of the melt is treated with 20% NaOH soln. to precipitate the sesquihydroxides and leave an excess of 3 to 4 ml, the soln. is heated, the ppt. is filtered off and washed with 2% NaOH soln., the filtrate is made 0·5 to 1 *N* in HCl, and W is pptd. by 1% mepacrine soln. The ppt. is collected, washed with a dil. soln. of mepacrine and then ignited. By pptn. from a strong acid medium the ppt. is purer than that obtained by use of amidopyrine or lactoacridine. With ores of low contents of W, the filtrate after removal of the hydroxide ppt. is reduced, and W is determined colorimetrically as SCN^-.
G. S. Smith

2183. **Salicylate method for determining microgram amounts of uranium.** P. F. Thomason, L. A. Nutting, U. Koskela and W. M. Byerly (Oak Ridge Nat. Lab., Oak Ridge, Tenn., U.S.A.). U.S. Atomic Energy Comm. ORNL-1641, 1955, 15 pp.—Uranyl ion (**I**) in amounts from 5 to 1800 μg can be determined spectrophotometrically. To a soln. of **I** (pH $\simeq$8) add excess of Na salicylate; measure the transmittancy at 360 mμ. Interference is caused by Fe, Th or PO_4^{3-}. Graphs are shown of transmittancy as a function of Na salicylate concn., pH, wavelength and **I** concn. for 10- and 25-ml cuvettes.
Chem. Abstr.

2184. **Colorimetric determination of uranium with dibenzoylmethane.** P. Blanquet (Minerais et Métaux, S.A., Paris, France). *Anal. Chim. Acta*, 1957, **16** (1), 44–56 (in French).—A spectrophotometric method for the determination of U is based on the formation of a coloured complex of UO_2^{2+} with dibenzoylmethane. It has been applied to minerals containing 0·05 to 25% of U. Certain interfering ions are first pptd. from the sample soln. with Na_2CO_3, $FeCl_3$ being added previously to ensure the pptn. of PO_4^{3-} and AsO_4^{3-}. The complex is formed in aq. pyridine in the presence of EDTA and tartaric acid and the extinction is determined at 415 mμ. The method can be used for samples that contain Mo, W, V, Ge, Th, F and P, but Sr, Ni, Au and Pt interfere when present in considerable excess.
W. C. JOHNSON

2185. **Determination of uranium in alloy systems.** G. W. C. Milner and J. W. Edwards (A.E.R.E., Harwell, England). *Anal. Chim. Acta*, 1957, **16** (2), 109–114 (in English).—Milligram amounts of U in soln. of alloys can be separated quant. from Fe, Cr, Cu, Ni, V, Ce, Nd and Bi by pptn. with $(NH_4)_2PO_4$ at 100° in the presence of a slight excess of 10% (w/v) aq. EDTA (disodium salt) and acetate buffer (≃pH 5). By dissolving the uranyl ammonium phosphate in hot 25% (v/v) HCl and passing the soln. through a lead reductor, the U^{4+} can be finally titrated with 0·02 *N* $Ce(SO_4)_2$ soln. and ferroin as indicator. This procedure, for which the lower limit is 0·5% of U, is particularly useful for Bi - U (1 to 20% of U) alloys; it does not permit separation from Sn^{2+}, Sn^{4+}, Mo and W.
W. J. BAKER

2186. **Separation of uranium from vanadium by ion exchange: with special reference to the determination of small amounts of uranium.** T. K. S. Murthy (Atomic Energy Establishment, Bombay, India). *Anal. Chim. Acta*, 1957, **16** (1), 25–28 (in English).—Uranium (down to 1 mg as U_3O_8) is separated from V (up to 1 g as V_2O_5) by means of a column of Amberlite IRA-400 anion-exchange resin. The resin is first converted to the chloride form by treatment with 2 *M* NaCl. The solution for treatment, which must be free from Cl^- and NO_3^-, contains 10 g of Na_2CO_3, VO_3^- equiv. to ⩾250 mg of V_2O_5, and $[UO_2(CO_3)_3]^{4-}$ equiv. to ⩾50 mg of U_3O_8 in 100 ml, and is passed through the column at a rate of 500 ml per hr. Elution with 30 ml of 10% Na_2CO_3 soln. at the same rate, followed by 200 ml at 100 ml per hr. and finally by 30 ml of water, removes the VO_3^-. The U is then eluted with 150 ml of 5% NaCl soln. containing 0·5% of Na_2CO_3. The V is determined by titration with a standard soln. of Fe^{2+} and the U by the NaOH - Na_2O_2 spectrophotometric method or gravimetrically.
W. C. JOHNSON

2187. **Indirect flame-photometric method for determination of halides.** O. Menis, H. P. House and T. C. Rains (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 76–79.—In this method, the accuracy of which is within ±5%, the sample soln. (containing from 0·02 to 0·2 milliequiv. of halide per litre) is added to a standard soln. of $AgNO_3$ containing from 5 to 50 μg of Ag per ml. After removal of silver halide by centrifugation, the luminosity due to residual Ag in the filtrate is measured by flame photometry at 328 mμ. The decrease in luminosity of the standard silver soln. is proportional to the concn. of halide in the sample. Calibration curves of luminosity *vs.* halide concn. are prepared previously. For the selective determination of I^- in the presence of Br^- and Cl^-, 0·5 *M* aq. NH_3 is added to the standard $AgNO_3$ soln., and for the determination of Br^- in the presence of Cl^-, 0·03 *M* aq. NH_3 is used as masking agent. There is no interference from U^{6+}, Cr^{6+} or PO_4^{3-} if the wt. ratios of these ions to halide are within the limits of 10^3 to 6×10^4, 2 to 20 and 20 to 50, respectively. The lower limits of detection are ≃0·5 μg of Cl^-, 1 μg of Br^- and 1·5 μg of I^- per ml. About 20 samples can be analysed in 1 hr.
W. J. BAKER

2188. **A direct microgravimetric determination of fluoride with triphenyltin chloride. (Two-phase precipitation.)** H. Ballczo and H. Schiffner (Chem. Lab., Univ. Vienna, Austria). *Mikrochim. Acta*, 1956, (12), 1829–1833 (in German).—An earlier method (*cf. Anal. Abstr.*, 1956, **3**, 3624) is adapted to micro techniques. From 10 to 150 μg of F may be determined in the presence of phosphate and borate ions with an accuracy of ≃ ±2%. Silicate and carbonate ions must be removed previously; Fe and Al, if present in quantity, are removed by pptn. with aq. NH_3, and Be and Zr interfere. To 2 ml of sample soln. (pH 4 to 8) in a micro filter-beaker add saturated triphenyltin chloride in $CHCl_3$ (2 ml, about 2·5 times the stoicheiometric quantity) and stir for 30 min. Filter, dry the ppt. at 110° and weigh.
P. S. STROSS

2189. **Argentimetric determination of chlorate.** A. Yu. Prokopchik and P. K. Norkus. *Trudy Akad. Nauk, LitSSR*, 1955, **B3**, 17–22; *Ref. Zhur., Khim.*, 1956, Abstr. No. 47,326.—The argentimetric determination of ClO_3^- is based on its reduction by $NaNO_2$ to Cl^-. Dissolve the sample of $KClO_3$ (0·3 g) in water (20 ml), add a 10% soln. of $NaNO_2$ (6 ml) and conc. HNO_3 (2 ml), and boil for 1 min. Cool, add 0·05 to 0·07 *N* $AgNO_3$ soln. (50 ml) and KNO_3 (1·5 to 2 g), boil for 3 min., cool to below 15°, add nitrobenzene (2 ml) and titrate with KSCN soln. in the presence of $Fe(NO_3)_3$. The mean error is −0·34%. If the AgCl ppt. is removed by filtration before the titration, the error is −0·07%. By potentiometric titration with $AgNO_3$ soln., the error is −0·44%. The consistently low results are explained as being due to loss of Cl during the reduction of the $KClO_3$.
C. D. KOPKIN

2190. **Current efficiency and titration efficiency in coulometric titrations with electrogenerated ceric ion. Determination of iodide.** J. J. Lingane, C. H. Langford and F. C. Anson (Harvard Univ., Cambridge, Mass., U.S.A.). *Anal. Chim. Acta*, 1957, **16** (2), 165–174 (in English).—Current efficiency for the electrogeneration of Ce^{4+} at a platinum anode can fall to 94 or 95% at both high and low c.d. The max. current efficiency is ≃99·8%. Results for the coulometric titration of I^- (1 to 30 mg) in 0·025 *M* $Ce_2(SO_4)_3$ and 0·9 *M* H_2SO_4 at c.d. from 2 to 19 mA per sq. cm are accurate to ≃ ±0·3%, even when the efficiency of generation of Ce^{4+} is only 98%. This is because I is oxidised to IO_3^- at a potential slightly greater than that at which Ce^{2+} are oxidised, and these transient IO_3^- oxidise I^- so that only a small fraction of the total amount of electricity arises from generation of Ce^{4+}. Titrations of I^- to I can be made satisfactorily in the absence of cerous salt only when the generating c.d. is less than the limiting c.d. for oxidation of I to IO_3^-. In titrations with electrogenerated Ce^{4+} (*cf. Anal. Abstr.*, 1956, **3**, 678; 2456), serious errors

can arise if the c.d. is above or below the optimum, especially when the ion being titrated, *e.g.*, Fe^{3+}, is incapable of direct oxidation at the platinum generator-electrode. W. J. BAKER

2191. **Identification of manganese.** R. Vanossi. *An. Asoc. Quim. Argentina*, 1956, **44** (2), 69–81.—A detailed discussion is given on the detection of Mn in the presence of interfering elements by attacking the sample with H_2SO_4 containing HNO_3 and $HClO_4$; heating to volatilisation with HCl and HBr; treatment with NaOH and Na_2O_2; extraction with ethyl acetate; pptn. of the Mn with aq. NH_3 in the presence of Br; solution in H_2SO_4; and oxidation with ammonium persulphate in the presence of Ag^+ and sodium hexametaphosphate to MnO_4. The sensitivity of the method is 0·8 to 2 μg with a 12-mg original sample. D. LEIGHTON

2192. **Colorimetric determination of manganese.** A. R. Tourky, I. M. Issa and I. F. Hewaidy (Univ. Cairo, Giza, Egypt). *Anal. Chim. Acta*, 1957, **16** (2), 151–154 (in English).—The clear-red colour formed when Mn^{2+} are oxidised to Mn^{4+} with H_2O_2 in the presence of telluric acid and 2·5 *M* NaOH (*cf. Anal. Abstr.*, 1956, **3**, 112) can be utilised for the determination of Mn in concn. from $\simeq 2$ to 8×10^{-4} *M*, for which Beer's law is valid. Only the theoretical equivalent of H_2O_2 should be added to the soln., which is heated at 60° for $\simeq 10$ min. to develop the colour; measurements are made in a Beckman spectrophotometer at 420 mμ in a 1-cm cell. The concn. of telluric acid should be 4 moles per mole of Mn^{2+}. The procedure is particularly applicable to biological liquids containing reducing organic materials. W. J. BAKER

2193. **Colorimetric determination of iron.** M. Grat-Cabanac (Fac. Sci. de Toulouse, France). *Anal. Chim. Acta*, 1957, **16** (2), 150 (in French).—In the colorimetric determination of trace amounts of Fe with 2:2′-dipyridyl or *o*-phenanthroline, the reduction of Fe^{3+} to Fe^{2+} can be effected with an excess of 20% aq. sodium hypophosphite at 80°. The reagent can be used in the visual colorimetric method (*cf. Anal. Abstr.*, 1955, **2**, 1218) because it does not reduce methylene blue or indigo carmine. W. J. BAKER

2194. **Colorimetric micro-determination of ferrous ions with dimethylglyoxime by the use of ascorbic acid as stabilising agent.** Tôru Hayashi and Akimasa Kajita (Takamine Lab., Sankyo Co. Ltd., Shinagawa-ku, Tokyo). *Ann. Rep. Takamine Lab.*, 1955, **7**, 130–132.—Ascorbic acid (**I**) stabilises the coloration of Fe^{2+} with dimethylglyoxime (**II**), and Beer's law is obeyed for $<0{\cdot}5$ mg of Fe per 100 ml. The sample soln. ($\simeq 1$ ml) containing 0·3 to 0·5 mg of Fe^{2+} is mixed with 2% **I** (5 ml) and **II** soln. (1% in 95% ethanol, 5 ml), adjusted to pH 11·3 with aq. NH_3 soln. and made up to 100 ml with 0·1 *N* aq. NH_3 soln. The extinction is measured at 530 mμ. The results compare favourably with those of methods in which hydroxylamine hydrochloride or hydrazine sulphate is used as stabiliser. K. SAITO

2195. **Benzenephosphinic acid as analytical reagent. Gravimetric determination of ferric iron.** J. E. Banks and D. A. Skoog (Univ., Stanford, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 109–113.—Milligram amounts of Fe^{3+} can be determined by quant. pptn. with a large excess of 0·2 *N* aq. benzenephosphinic acid in 0·1 to 1 *N* H_2SO_4 or HCl at 90° to 100°. The granular ppt. of ferric benzenephosphinate is digested (steam bath) for $\simeq 1$ hr. and dried at 110° to const. wt. (3 phosphinate ions $\equiv$ 1 Fe^{3+}). The ppt. is more sol. than $Fe(OH)_3$ and losses of Fe can amount to 0·1 to 0·5 mg, equiv. to an error of 0·1 to 0·3% for concn. of Fe >60 mg. Max. accuracy is attained when the sample contains >100 mg of Fe. W. J. BAKER

2196. **Cerimetric determination of iron in iron salts.** K. A. Smirnova. *Trudy Vses. Nauch. Inst. Khim. Reaktivov*, 1956, (21), 52–54; *Ref. Zhur., Khim.*, 1956, Abstr. No. 54,774.—After reduction of the sample (ferric oxide or ferric nitrate) with $SnCl_2$ by the standard method, 0·1 to 0·2 g of Na_2CO_3 is added to remove air from the flask, and then one drop of molybdosilicic acid soln. (Faĭnberg *et al.*, *Zavod. Lab.*, 1945, **11**, 381) is added and the hot soln. is titrated with 0·1 *N* $Ce(SO_4)_2$ until a green colour appears. The cooled soln. is then treated with 100 ml of dil. H_2SO_4 (1 + 2) and 0·3 ml of 1% phenylanthranilic acid soln., and titrated until the colour changes to brown. From the total vol. of the $Ce(SO_4)_2$ soln. used, the amount expended in oxidising the excess of $SnCl_2$ is deducted. The results obtained by this method and the cerimetric method in which $HgCl_2$ is used are in close agreement. G. S. SMITH

2197. **Oxidimetric titration with potassium ferricyanide solution. Determination of iron and titanium.** Mitsuo Kiboku (Fac. Sci. and Tech., Kinki Univ., Fuse, Osaka). *Japan Analyst*, 1956, **5** (9), 503–505.—The oxidimetric titration of Fe^{2+} and Ti^{3+} with $K_3Fe(CN)_6$ was studied in a basic soln. containing Na_3PO_4 (**I**) or $Na_4P_2O_7$ (**II**), with cacothelin as indicator. Ferrous iron is satisfactorily titrated at pH 4·3 to 10·0, in the presence of either **I** or **II**; Ti^{3+} are titrated at pH $<11{\cdot}4$ and at $<10{\cdot}2$, by the use of **I** and **II**, respectively. Their sum is determined in a weakly basic soln. in the presence of **I** or **II**. Titanium is titrated in an acid soln., the formation of Turnbull's blue being used as an indication of the end-point; Fe is calculated by difference. Zinc (dissolved from zinc amalgam used as reducing agent) does not interfere. K. SAITO

2198. **Benzenephosphinic acid as analytical reagent. Separation of ferric iron from certain anions and cations.** J. E. Banks, J. F. Pennell and D. A. Skoog (Univ., Stanford, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 113–116.—Iron can be separated almost completely from relatively large amounts (40 to 600 mg per 60 mg of Fe) of Mn^{2+}, Ni^{2+}, Co^{2+}, Cd, Mg, alkaline-earth metals, As^{3+}, As^{5+}, Cl^-, PO_4^{3-}, citrate, tartrate and oxalate by pptn. with 0·2 *N* aq. benzenephosphinic acid in 0·5 *N* H_2SO_4 (*cf. Anal. Abstr.*, 1957, **4**, 2195). Owing to co-pptn., separation from Cr, V and Al is incomplete; the presence of Cu^{2+} also causes interference. Iron cannot be separated from Ti, Mo, Zr, Ce, U, W, Sn, Bi, Ag and Hg because these elements form insol. ppt. with benzenephosphinic acid. Separation of Fe from Zn by this method is adequate only when the concn. of the two elements are approx. equiv. W. J. BAKER

2199. **Ion-exchange studies of phosphates. II. Phosphoric acid as an eluent in the column separation of tervalent iron from bivalent copper and manganese.** J. A. R. Genge and J. E. Salmon (Chem. Dept., Battersea Poly., London). *J. Chem. Soc.*, 1957, 256–262.—The separation of macro

quantities of Fe^{3+} from Cu^{2+} and Mn^{2+} is achieved through the preferential complex formation between Fe and phosphate. The optimum conditions were established by batch experiments with Zeo-Karb 225 converted to its metal forms (Fe^{3+}, Cu^{2+} and Mn^{2+}), and by column experiments with the hydrogen form of the resin. The factors studied were pH, temp., time of contact, resin-particle size and the degree of cross-linking of the resin. In the pH range 0·2 to 1·2 (with pH 0·75 as optimum), H_3PO_4 is the most effective of the series of acids studied for removing Fe from the resin, whereas it has least effect on Cu and Mn. An increase of temp. from 20° to 40° has little effect on the amounts of the three metals removed, but reduction in particle size of the resin from 22 to 30 mesh to 40 to 80 mesh showed an increased rate of exchange of Fe^{3+} for H^+, and a smaller increase occurred for the Cu^{2+} and Mn^{2+}. Variations in the amount of cross-linking of the resins had little effect. G. S. ROBERTS

2200. **Separation of iron from aluminium by precipitation of ferric periodate from homogeneous solution.** L. Ginsburg, K. Millar and L. Gordon (Univ., Syracuse, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 46–48.—Milligram amounts of Fe can be quant. separated from similar amounts of Al by pptn. from homogeneous soln. as periodate, in the presence of acetamide and in dil. HNO_3 at final values of pH of 1·2 to 1·7. Co-pptn. of Al is negligible (Gordon and Ginsburg, *Anal. Abstr.*, 1957, **4**, 2201). The ppt. is dense, easily filtered and washed, and can be ignited slowly to 1000° for weighing as Fe_2O_3. One pptn. will separate ≃85 mg of Fe from ≃10 mg of Al, but two pptn. are needed to separate the same amount of Fe from ≃100 mg of Al. Interfering ions are Ti^{4+}, Hf^{4+}, Zr^{4+}, Zn^{2+} and Cu^{2+}, which are also pptd.
W. J. BAKER

2201. **Co-precipitation studies with ferric periodate precipitated from homogeneous solution.** L. Gordon and L. Ginsburg (Univ., Syracuse, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 38–46.—The co-pptn. of Al, Zn and Y has been studied (radiometrically for Zn and Y) with ferric periodate (pptd. from homogeneous soln. by hydrolysis of acetamide at ≃80°) as carrier. Co-pptn. of Al and Y is slight (0·4 to 1·2% for Al, ≃0·3% for Y) until 99·9% of the Fe has been pptd.; the amount of Y co-pptd. is independent of its initial concn. within a fourfold change. Zinc is co-pptd. in appreciable amounts before the end of the pptn. of Fe, the amount depending on the initial concn. of Zn ($1·4 \times 10^{-5}$ to $3·5 \times 10^{-3}$ *M*). The Freundlich adsorption isotherm is approx. valid for the co-pptn. of Zn. In all instances, occlusion during the formation of the ppt. is negligible. The data are of use in the prep. of relatively pure carriers. Analysis indicates that the composition of the ferric periodate is probably $Fe(IO_4)_3.2Fe_2O_3.7H_2O$. W. J. BAKER

2202. **Colorimetric method for the determination of magnesium in cast iron.** A. T. Pilipenko and L. I. Dubovenko. *Nauk. Zap. Kiev Univ.*, 1955, **14** (4), 143–151; *Ref. Zhur., Khim.*, 1956, Abstr. No. 47,271.—Magnesium is separated as the 8-hydroxyquinolinate, the ppt. is dissolved in conc. HCl, and 8-hydroxyquinoline is determined colorimetrically by the colour of the complex with Fe^{3+} in CCl_4. Manganese and iron interfere. The former is removed by the persulphate method in alkaline medium, and the latter on a mercury cathode, or by the quicker and more convenient method of extraction of the chloride or ethylxanthate complex of Fe^{3+}. In the latter case only the main mass of iron is removed. C. D. KOPKIN

2203. **Rapid spectrophotometric determination of a small amount of lead in iron and steel.** Shigeo Wakamatsu (Toto Steel Mfg. Co. Ltd., Minamisunamachi, Koto-ku, Tokyo). *Japan Analyst*, 1956, **5** (9), 509–512.—The extraction of lead dithizonate from an $HClO_4$ soln. of steel was studied in the presence of CN^- and SO_3^{2-}. The interference of Fe (<0·1 g) is eliminated so that the separation of Pb (0·0005 to 0·2%) by pptn. as PbS becomes unnecessary. The use of benzene as solvent makes the extraction much easier. The time taken for an estimation is ≃25 min. *Procedure*—The sample (1 g) is dissolved in HNO_3 (1 + 1, 10 ml) and $HClO_4$ (60%, 10 ml), heated to white fumes, cooled and diluted to 250 ml. (When WO_3 is pptd., it is filtered off.) A 5 to 25-ml portion is diluted to 40 ml, treated with tartaric acid (20%, 2 ml), Na_2SO_3 (saturated soln., 10 ml) and KCN (20%, 10 ml) and heated at 80°. The product is cooled, made basic with aq. NH_3 soln. (sp. gr. 0·9, 10 ml) and shaken with dithizone soln. (0·01% in benzene, 10 ml). The benzene layer is washed with dil. aq. NH_3 soln. (1 + 100, ≃50 ml) containing saturated Na_2SO_3 soln. (2 ml) and the extinction is measured at 520 mμ. K. SAITO

2204. **Rapid photocolorimetric method for estimating sulphur in cast iron.** G. Ciuhandu and A. Dewald. *Stud. Cercet. Chim., Bucharest*, 1955, **3** (3–4), 293–300.—The sample of cast iron is attacked with conc. HCl, and the H_2S evolved is passed into a 0·2% soln. of *p*-aminodimethylaniline. On treating the soln. with $FeCl_3$, the characteristic colour of methylene blue is developed, and the extinction of the soln. is measured. For the preparation of the reagent, the use of the more stable *p*-acetamidodimethylaniline is recommended. J. H. WATON

2205. **Improvements in or relating to methods of testing in metallurgical processes.** Westfalenhütte A.-G., Dortmund-Hörder Hüttenunion A.-G. and A. Thyssenhütte A.-G. Brit. Pat. 754,384; date appl. 29.3.54. Germany, date appl. 30.3.53.—Quant. changes in the concn. of constituent elements in metal baths are determined by adding radioactive isotopes of the element to be determined to the melt and measuring their radiation intensities, by means of a Geiger counter, during the melting. Thus P is determined in pig iron or steel by adding the β-emitting isotope ^{32}P (≃1 mC per ton of metal), *e.g.*, in the form of red phosphorus which has been partly converted into ^{32}P by irradiation, or as iron phosphide prepared from such irradiated red phosphorus, to the molten metal.
J. M. JACOBS

2206. **Non-destructive microchemical method of sorting steels.** W. R. Nall and R. Scholey (Bragg Lab., N.O.I.D., Sheffield, England). *Metallurgia, Manchr*, 1956, **54**, 97–102.—The procedure described, for the rapid sorting of carbon and low alloy steels, is based on the estimation of Mn, Ni, Cr and Mo with simple, inexpensive apparatus. A current is passed, by means of a modified torch-like assembly, through a drop of dilute acid on a filter-paper in contact with the steel, and colours characteristic of the alloying elements are developed by appropriate reagents. These colours are compared with known standards similarly prepared. For Mn, Ni and Mo, dil. HCl is used as electrolyte, the papers

are dried in a miniature oven and periodate - 4:4′-tetramethyldiaminodiphenylmethane, alkaline dimethylglyoxime, and NH_4SCN - $SnCl_2$ are adopted as the respective colouring agents. For Cr, a 40% soln. of conc. HNO_3 is employed as electrolyte and $AgNO_3$ soln. as spraying agent. A series of results of sufficient accuracy for identification to known specifications is reported, and it is suggested that the scope of the procedure could be widened by including tests for other elements. J. O. LAY

2207. **Determination of small amounts of cobalt.** I. G. Shafran and R. I. Gordeeva. *Trudy Vses. Nauch. Inst. Khim. Reaktivov*, 1956, (21), 26–32; *Ref. Zhur., Khim.*, 1956, Abstr. No. 54,777.—To determine Co ($<10^{-5}$ %) in chemical reagents, CoO_2, obtained by oxidation with $KMnO_4$, is co-pptd. with MnO_2, the ppt. is dissolved in 1 ml of conc. HCl containing 0·1 ml of 30% H_2O_2, and the soln. is evaporated to dryness. The residue is dissolved in water, 0·5 ml of 0·1% nitroso-R salt soln. and 1 ml of saturated Na acetate soln., which has been neutralised to pH ≃8·5, are added, the soln. is boiled for 2 min., 1 ml of dil. HNO_3 (1 + 1) is added, and the soln. is then evaporated to a vol. of 3 to 4 ml. The colour is compared with that of standards prepared similarly. The error is ±0·05 μg of Co. G. S. SMITH

2208. **The absorptiometric determination of traces of cobalt with 1-nitroso-2-naphthol.** G. Middleton and R. E. Stuckey (B.D.H. Ltd., City Rd., London, England). *Clin. Chim. Acta*, 1956, **1** (2), 135–142.—Interfering metals are removed by means of an alumina column; Fe is removed after reduction with alkaline hydroxylamine; the Co in the complex is determined by reading the extinction at 415 mμ in $CHCl_3$ soln. Methods are described for purifying the reagent, assuring suitability of the Al_2O_3, and extraction of Co from biological materials after destruction of organic matter. H. F. W. KIRKPATRICK

2209. **Determination of metal ions by means of their oxalates. VII. Determination of cobalt.** Kyozo Kawagaki (Fac. of Engng, Niigata Univ., Nagaoka). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1956, **77** (10), 1461–1464.—The pptn. of Co^{2+} with oxalic acid is not quant. even when >20 times its weight of the Co is used. When the same vol. of acetic acid is added to the sample soln. (<0·05 *N* acid, neutral or <0·5 *N* basic), 5 to 150 mg of Co is quant. pptd. The ppt. is washed with water, dissolved in dil. H_2SO_4, and titrated with $KMnO_4$. K. SAITO

2210. **New rapid and accurate method for the estimation of cobalt in the presence of iron and aluminium, and for the simultaneous estimation of cobalt and nickel in the presence of iron and aluminium.** G. Spacu and M. Schiau (Academ. R.P.R., Bucharest). *Stud. Cercet. Chim., Bucharest*, 1955, **3** (3–4), 167–173.—Cobalt is quant. pptd. as the complex $[CoPy_4(SCN)_2]$ in the presence of Fe and Al, if the Fe and Al are masked with Na sulphosalicylate. Ammonium salts must be removed by evaporation. In the same way, both Co and Ni can be separated from Al and Fe and, after weighing the mixture of the complexes, the Ni is determined with dimethylglyoxime. J. H. WATON

2211. **Determination of small amounts of cobalt in steels and nickel alloys by the isotope dilution - anodic deposition method.** D. Salyer and T. R. Sweet (Ohio State Univ., Columbus, U.S.A.). *Anal. Chem.*, 1957, **29** (1), 2–4.—Separation as cobaltinitrite followed by isotope dilution - anodic deposition (*cf. Anal. Abstr.*, 1956, **3**, 1732) enables <1% of Co to be determined with a max. error of 0·02% and a standard deviation varying from 0·005% (for 0·14% of Co) to 0·025% (for 0·47% of Co). A known wt. of ^{60}Co is added to the sample soln., any SiO_2 is removed, and the Fe (if >5%) is extracted (twice) into diethyl ether from a 6 to 8 *N* HCl soln. An excess of 30% NaOH is added to the aq. phase (concentrated by evaporation), the ppt. is dissolved in the min. of acetic acid (any MnO_2 is filtered off), and the Co is then pptd. with 50% aq. KNO_2 at 100° (two pptn. may be necessary). After 30 min. the cobaltinitrite ppt. is filtered off and dissolved in dil. H_2SO_4, the soln. is buffered to pH 7·8 (0·1 *M* H_3BO_3 - 0·05 *M* K_2SO_4 plus 0·5 *M* KOH) and is then electrolysed for 40 min. at 20° and 1·5 to 1·8 V. The deposit (1 to 2 mg) on the platinum-disc anode is weighed as cobaltic oxide trihydrate and its sp. activity is determined (20-min. count, aluminium screen). The percentage of Co is calculated from a standard linear curve. Since the isotope-dilution method does not require isolation of all the Co, the ageing of the cobaltinitrite ppt. can be reduced to ≃30 min.; the vol. of the soln. is also kept at a min. to increase the rate of pptn. W. J. BAKER

2212. **Chromatographic separation of Ni^{2+} and Co^{2+}.** V. V. Oshchapovskiĭ (Lvov Polytech. Inst.). *Zhur. Anal. Khim.*, 1956, **11** (5), 606–612.—Dimethylglyoxime is used as adsorbent in a chromatographic column. The reaction with Ni^{2+} is most sensitive at a pH of 5 to 6. The presence of Co^{2+} reduces the sensitivity, but 0·5 μg of Ni in one drop of soln. can be detected in the presence of 5000 times as much Co. To determine Ni in salts of Co, the sample is dissolved in a buffer soln. of pH 6 to 10 so that the content of Ni is ≃10 to 20 μg per ml, and 2 to 5 ml of the soln. is passed through a layer 15 to 20 mm high of moist dimethylglyoxime at a rate of ≃1 ml per min. The column is then washed with several portions (2 to 3 ml) of water until the filtrate is colourless and gives no reaction for Co. The Ni is dissolved by washing with 0·1 *N* HCl and water and determined colorimetrically in the filtrate by means of $(NH_4)_2S_2O_8$ and dimethylglyoxime. G. S. SMITH

2213. **New gravimetric methods for the rapid estimation of the elements nickel, cobalt and cadmium.** D. Pirtea, G. Dumitrescu and N. Melencu. *Stud. Cercet. Chim., Bucharest*, 1955, **3** (3–4), 237–242.—Nickel, Co and Cd are each pptd. quant. as the complex $[MPy_4][Cr_2O_7]$ by the addition of pyridine and $K_2Cr_2O_7$ soln. at room temp. After filtration, the ppt. are washed with ethanol and with ether, and are dried in a vacuum desiccator. Each analysis can be performed in 45 to 50 min. J. H. WATON

2214. **Determination of ruthenium by potentiometric titration with titanous chloride.** N. K. Pshenitsyn and S. I. Ginzburg. *Izv. Sektora Platiny, I.O.N.Kh., Akad. Nauk, SSSR*, 1955, (32), 20–30; *Ref. Zhur., Khim.*, 1956, Abstr. No. 47,307.—In the titration of soln. of $RuCl_4$ (**I**) or $K_2[RuOHCl_5]$ (**II**) in 0·3 to 1 *N* HCl by a 0·01 to 0·02 *N* soln. of $TiCl_3$ (**III**) in 10% HCl, the reduction of Ru^{4+} to Ru^{3+} passes through an intermediate complex, the formation of which requires 0·5 equiv. of **III**. With the formation of the complex (which, it is

suggested, contains Ru^{IV} and Ru^{III}), there is a considerable change of potential on the potentiometric titration curve. If the reduction is carried out not at 18° to 20° but at 70° to 80°, after the first sudden change of potential there is a further reduction, and when the reduction of Ru^{IV} to Ru^{III} is complete there is a second sharp change of potential. From spectrophotometric data at 380 mμ, equilibrium at room temp. before the first change of potential is established within a few min., and after the first change of potential within 2 to 3 hr. Therefore in the proposed method of determining Ru, the titration is carried out at room temp. to the first change of potential. Before titrating, the Ru in 1·5 to 2 *N* HCl is oxidised to the stable **I** by boiling with a small excess of chlorine water for 10 min. In determining Ru in soln. in which, as a result of long standing, partial reduction or more complete hydrolysis of **II** has taken place, the soln. are first evaporated 2 or 3 times with conc. HCl and then oxidised by boiling with chlorine water. After removal of excess of Cl, the cooled soln. is diluted to 0·7 to 0·8 *N* in HCl, and titrated in CO_2 by a soln. of **III**, with an indicating platinum electrode. To obtain sharp changes of potential, the soln. must contain ⩾20 mg of Ru in 100 ml. The error is then ⩾4·8%. By titrating at 60° to 80° it is possible to determine Ru by the second change of potential, although the accuracy is then less. C. D. KOPKIN

2215. **Gravimetric determination of palladium(II) with *p*-ethylsulphonylbenzaldehyde thiosemicarbazone.** Sumio Komatsu, Teruo Kida and Zin Hiroaki (Fac. Liberal Arts, Toyama Univ., Toyama). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1956, **77** (9), 1437–1438.—The reagent (**I**) in 5 *N* HCl yields an orange - yellow ppt. with Pd^{II}, which can be washed with 1% HCl and water and dried at 110° to 120° to const. weight. The pptn. is quant. in <0·5 *N* HCl; the amount of the ppt. decreases with increasing concn. of HCl. An excess of **I** is sparingly sol. in dil. HCl and results in high values; the vol. of the sample soln., from which the ppt. is formed, should be >300 ml for <10 mg of Pd. Similar ppt. are formed with Au, Ag, Cu, Pt and Hg. K. SAITO

2216. **Spectrophotometric determination of iridium with leuco-crystal violet.** G. H. Ayres and W. T. Bolleter (Univ. of Texas, Austin, U.S.A.). *Anal. Chem.*, 1957, **29** (1), 72–75.—From 0·5 to 4 p.p.m. of Ir can be determined spectrophotometrically by measuring the extinction, at 590 mμ in a 1-cm cell, of the coloured dye formed by oxidation of leuco-crystal violet (4:4′:4″-hexamethyltriaminotriphenylmethane) at pH 3·5 to 4·7 (acetate buffer). The colour develops quickly at room temp. and is stable for ⩽12 hr.; the specific absorptivity (extinction ÷ p.p.m. of Ir) = 0·25. The sample soln. should be initially taken to fuming with HNO_3, $HClO_4$ and H_3PO_4, and adjusted finally to 1 *M* in H_3PO_4. The colour reagent is a 0·1% soln. of leuco-crystal violet in 1 *M* H_3PO_4. The error is ≃0·6%; the mean deviation is 0·003 for 2 p.p.m. of Ir. Removal of Au^{3+} (by amalgamation after reduction with quinol) is necessary because it oxidises the leuco-dye. W. J. BAKER

2217. **Benzyldimethylphenylammonium chloride: a reagent for the gravimetric determination of platinum.** D. E. Ryan (Dalhousie Univ., Halifax, Nova Scotia, Canada). *Canad. J. Chem.*, 1956, **34** (12), 1683–1686.—Platinum is quant. determined by pptn. from $PtBr_6^{2-}$ soln., in conc. HBr, with the reagent benzyldimethylphenylammonium chloride, 5 ml of a 5% aq. soln. being added for each 10 mg or less of Pt present. The pptd. bromoplatinate is filtered off, washed with 0·1% reagent soln., dried at 110°, and weighed. Commonly associated base metals do not interfere. O. M. WHITTON

2218. **Polarographic determination of base-metal impurities in platinum.** B. A. Muromtsev and V. D. Ratnikov. *Izv. Sektora Platiny, I.O.N.Kh., Akad. Nauk, SSSR*, 1955, (32), 52–58; *Ref. Zhur., Khim.*, 1956, Abstr. No. 47,341.—The polarographic determination of Fe, Ni, Pb, Cu and Te in platinum is carried out by dissolving the sample in aqua regia, evaporating three times with conc. HCl, dissolving the dry residue in 2% HCl, and removing Pt from these base metals by a previously described method of reduction with HgCl (Karpov and Fedorova, *Izv. Inst. Platiny, Akad. Nauk, SSSR*, 1935, **12**, 163). The $HgCl_2$ formed in the reduction is converted into mercury chloramide by the action of NH_3, and is removed by ignition. Dissolve the dry residue in a known vol. of dil. HCl and determine Pb in an aliquot on a background of 0·5 *M* K Na tartrate. In another aliquot, remove Fe by two pptn. with aq. NH_3 and determine Cu and Ni on a background of *M* aq. NH_3 and 0·2 *N* NH_4Cl. Dissolve the $Fe(OH)_3$ in hot dil. HCl, add a crystal of ammonium oxalate, evaporate till HCl is removed, dissolve the residue in a saturated soln. of ammonium oxalate or *M* K oxalate and subject to polarography. The polarograms are taken as follows—for Cu and Ni from −0·2 to −1·4 V; for Fe from 0 to −0·6 V; for Pb from −0·3 to −0·9 V. Determine Te in a separate sample by mixing with finely divided NaCl and active carbon and chlorinating for 5 hr. at 700°. The sublimed Te is trapped in 50% HCl and separated as elementary Te by the action of SO_2 and hydrazine. Evaporate and ignite to remove the H_2SO_4 formed, oxidise Te in the residue with conc. HNO_3, and again evaporate to dryness. Dissolve the residue, and obtain a polarogram in a solution of 0·3 *M* aq. NH_3 with 0·1 *N* NH_4Cl, or *M* NaOH, from −0·5 to −1·1 V. C. D. KOPKIN

See also Abstracts 2059, 2061, 2062, 2063, 2064, 2067, 2068, 2071, 2072, 2074, 2075, 2080, 2220, 2249, 2264, 2278, 2288, 2387, 2412, 2430, 2443, 2471, 2472.

3.—ORGANIC ANALYSIS

2219. **Alcoholic hexanitratoammonium cerate as a quantitative oxidant for organic substances.** D. G. M. Diaper and F. R. Richardson (Royal Military College, Kingston, Ontario, Canada). *Canad. J. Chem.*, 1956, **34** (12), 1835–1837.—The method is based on the interaction of an alcohol, *e.g.*, methanol or ethanol, and hexanitratoammonium cerate, and the bleaching of the orange colour produced with an organic compound such as anethole, hydroxylamine, oxalic acid, and the oximes of *cyclo*hexanone, *cyclo*heptanone, heptaldehyde and butyraldehyde, but not vanillin which gives a yellow end-product. A 0·1 to 0·3-*M* aq. soln. of the cerate is employed, freshly prepared each day. By using a spot-plate technique, as little as 10 μg can be detected. In the volumetric technique, the substance dissolved in methanol is

titrated against aq. cerate from a burette until at the end-point a yellow - orange colour is produced. Preferably, aq. HNO_3, to a final concn. of 0·1 *N* to 0·2 *N*, is added as catalyst. A colorimetric technique is also described. Because the alcohol - cerate colour does not develop instantaneously and because it slowly fades, the soln. should stand in the dark for at least 15 to 20 min. after being mixed. O. M. WHITTON

2220. **A micro-method for the detection of carbon in organic and inorganic compounds by magnesium fusion.** M. Jureček and M. Nepraš (Inst. for Anal. Chem., Tech. High School for Chem., Pardubice, Czechoslovakia). *Mikrochim. Acta*, 1956, (12), 1762–1766 (in German).—The sample (1 to 2 mg) is fused with magnesium powder in a combustion tube, any carbon being converted into carbide. The fused mass is ground and transferred to a flask fitted with two absorption tubes, one filled with alkaline H_2O_2, and the second with pumice soaked in $HgCl_2$ soln. to absorb H_2S, phosphine, arsine, and boron and silicon hydrides. Dilute acid is added to the fused mass, and the gas, after passing through the absorption tubes, is bubbled into an ammoniacal $CuSO_4$ - hydroxylamine hydrochloride soln. Any acetylenic compounds, formed from the magnesium carbide, are absorbed and give a blue coloration. More than 50 organic and 15 inorganic compounds, including some containing H, O, N, halogens, S, P and As, were tested and gave a positive reaction. P. S. STROSS

2221. **The direct determination of oxygen in fluorine-containing organic compounds.** L. Mázor (Inst. Gen. Chem., Tech. Univ., Budapest, Hungary). *Mikrochim. Acta*, 1956, (12), 1757–1761 (in German).—The determination, by the Unterzaucher method, of oxygen in compounds that contain F gives high results, since any HF which is formed reacts with the silica of the combustion tube to give water. This reacts with the heated carbon to give CO. To overcome this, a layer of magnesium nitride packed into position with silver wool is introduced into the combustion tube, and the sample is distilled on to this layer. The HF combines with the Mg_3N_2 to give MgF_2, which is stable. P. S. STROSS

2222. **Organic elementary analysis. II. Determination of nitrogen in refractory compounds by modified micro-Dumas method.** Minoru Fukuda (Fac. Pharm., Osaka Univ., Hotarugaike, Toyonaka, Osaka). *J. Pharm. Soc. Japan*, 1956, **76** (9), 1041–1044.—Refractory compounds such as sulphathiazole, thiothiamine or 2-*p*-aminophenyl-1:4:8-pyrimidazole give low values in the determination of N by the micro-Dumas method. Satisfactory results are obtained, however, by covering the sample in the quartz boat with NiO, obtained by the ignition of $NiCO_3$ or $Ni(NO_3)_2$. The temp. of the furnace is kept at >800°. K. SAITO

2223. **Micro-determination of fluorine in organic compounds.** T. S. Ma and J. Gwirtsman (Brooklyn College, Brooklyn, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 140–142.—The organic fluoro compound is decomposed by sodium fusion in a Parr bomb and the product is taken up with water. The F is separated as H_2SiF_6 by steam-distillation from $HClO_4$ and is determined by titration with $Th(NO_3)_4$; a visual colorimetric or a photo-electric end-point is used. Results were within ≃ ±3% of theoretical values when applied to pure fluoro compounds. Other halogens, N, S and P do not interfere. G. P. COOK

2224. **Modern developments in organic elementary micro-analysis. III. Chlorine, bromine, iodine, fluorine, sulphur.** G. Kainz (II Chem. Lab., Univ., Wien). *Öst. ChemZtg*, 1957, **58** (1–2), 8–13.—A review with 79 references. (*Cf.* Kainz, *Öst. ChemZtg*, 1956, **57**, 216, 242.) A. R. ROGERS

2225. **The micro-determination of sulphur in organic compounds.** A. S. Inglis (Org. Chem. Dept., Univ., Liverpool, England). *Mikrochim. Acta*, 1956, (12), 1834–1838 (in English).—The sample is fused with Na_2O_2 in a nickel bomb, the soln. of the fused mixture is treated with a cation-exchange resin, and the sulphate is titrated by the method of Fritz and Yamamura (*Anal. Abstr.*, 1956, **3**, 389). To the sample (3 to 10 mg) in a small nickel bomb add Na_2O_2 (0·8 g) and ethanediol (two drops) and heat to redness for 2·5 min., cool and open the bomb, place in a small beaker and cover with water. Add a platinum-black catalyst, boil gently for 10 min., cool to room temp. and dilute to 50 ml. Transfer the alkaline soln. to a beaker, add Amberlite IR-120 (H) resin (27 g) and stir for 5 min. To an aliquot (25 ml) add ethanol (100 ml), aq. thoron indicator soln. (0·2%) (three drops) and titrate with 0·01 *N* $Ba(ClO_4)_2$ to a permanent pink colour. The method is adapted to the simultaneous determination of S and Cl, and to the determination of S in the presence of F. P. S. STROSS

2226. **Organic quantitative analysis. IX. Micro-determination of sulphur in compounds containing barium, lead, silver or magnesium.** M. Večeřa and L. Synek (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1957, **51** (1), 171–173.—The formation of Ba, Pb, Ag or Mg salts of thiols or sulphonic acids is often used for the identification of these compounds. When determining S in these salts, with a combustion method, the sulphur oxides must be quant. removed; the addition of V_2O_5 and the use of a temp. of 1200° to 1300° have been found suitable. J. ZÝKA

2227. **Determination of small amounts of alkoxyl groups in organosilicon compounds.** S. V. Syavtsillo and E. A. Bondarevskaya. *Zhur. Anal. Khim.*, 1956, **11** (5), 613–614.—The method of Kreshkov and Nessonova (*Zhur. Anal. Khim.*, 1949, **4**, 220) is modified so that small amounts (<0·01%) of ethoxy and butoxy groups in ethylphenylpolysiloxanes can be determined with an error of ±10% of the content. For ethoxy groups an ampoule containing the sample (≃0·02 g) is broken under 3 ml of HI (sp. gr. 1·69 to 1·70) in a reaction vessel in a stream of CO_2, and the gases are passed through a washing vessel containing a 10% soln. of a mixture (1 + 1) of $CdSO_4$ and $Na_2S_2O_3$ and into a receiver containing 4 ml of a 10% soln. of Na acetate in glacial acetic acid and five to six drops of Br. The reaction mixture is boiled gently for 45 min. After this, the contents of the receiver are poured into a flask containing 1 g of Na acetate and treated with a few drops of formic acid (to destroy free Br) and then with 2 ml of dil. H_2SO_4 (1 + 4) and 1 ml of 10% KI soln. The liberated iodine is titrated with 0·02 *N* $Na_2S_2O_3$. For butoxy groups the reaction mixture is heated at 40° for 30 min., then at 60° for 30 min. and then at 100° for 30 min. G. S. SMITH

2228. **The analysis of halogenated hydrocarbons by vapour-phase chromatography.** F. H. Pollard and C. J. Hardy (The Univ., Bristol, England). *Anal. Chim. Acta*, 1957, **16** (2), 135–143 (in English).—The qual. and quant. separation of <12 μl of mixed halogenated hydrocarbons by gas - liquid partition chromatography with different stationary phases (dibutyl or dinonyl phthalate, silicone fluid) and column temp. (20° to 100°) are described, with special reference to the selection of operating conditions for the accurate analysis of mixtures. The separated compounds can be identified from curves of log (corrected retention vol. per g of stationary phase) *vs.* (column temp.)$^{-1}$, as described by Littlewood *et al.* (*J. Chem. Soc.*, 1955, 1480). Factors affecting the accuracy when an internal standard, *e.g.*, acetone, is used are discussed and the degree of error and reproducibility of the values obtained by three methods of calibration are compared for a mixture of three chloromethanes separated on a column of dinonyl phthalate on celite at 40° with N as carrier gas and the katharometer held at 36°. In general, the error is $<2\%$, but it rises to 4% for the lower amounts [$\simeq 10\%$ (v/v)] of each component. The use of He in place of N leads to improved accuracy. The heats of soln. in the liquid phase and the corresponding latent heats of vaporisation are listed for several chlorinated hydrocarbons. W. J. Baker

2229. **Quantitative determination of fluorinated hydrocarbons by gas chromatography.** W. C. Percival (E.I. du Pont de Nemours & Co., Inc., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 20–24.—Commercial mixtures (Freons) of trichlorofluoromethane, dichlorodifluoromethane and 1:2-dichlorotetrafluoroethane were successfully analysed by gas chromatography. The 95% confidence limits for the average of duplicate determinations is $\simeq \pm 0{\cdot}5\%$ at the 50% level for any component and the accuracy is also within this limit. The effect of variables in the method has been quant. measured. G. P. Cook

2230. **Ethanol.** British Standards Inst. (2, Park St., London). B.S. 507:1956, 9 pp.—The specification includes standards and methods of determination for strength, residue on evaporation, miscibility with H_2O, alkalinity or acidity and content of aldehydes and ketones. Data tabulated show the relation between proof strength, sp. gr. in air and percentage of ethanol by wt. and by volume.
O. M. Whitton

2231. ***n*-Butanol.** British Standards Inst. (2, Park St., London). B.S. 508:1956, 9 pp.—The specification includes standards and methods of determination for specific gravity at 15·5°/15·5°, 20°/20°, and 25°/25°; distillation range; flash pt.; H_2O content; residue on evaporation; acidity; and content of aldehydes and ketones.
O. M. Whitton

2232. **Analysis of mixtures of glycols by ion-exchange chromatography.** R. Sargent and W. Rieman, III (Rutgers Univ., New Brunswick, N.J., U.S.A.). *Anal. Chim. Acta*, 1957, **16** (2), 144–148 (in English).—A mixture of diethylene glycol, ethanediol, propane-1:2-diol, the *meso* and the (±) isomers of butane-2:3-diol, and glycerol can be separated quant. by anion-exchange chromatography on two separate columns (76·5 cm × 2·28 cm, and 20 cm × 2·28 cm) of Dowex 1-X8 (200 to 300 mesh, H form) at 25°. An aliquot of 2 ml ($\equiv$ >10 mg of any one glycol) is passed through one column and a similar aliquot through the other at a rate of 0·5 cm per min. The longer column is then eluted with 0·925 *M* $NaBO_2$ and the shorter one with 0·02 *M* $Na_2B_4O_7$, the two eluates being collected in seven and five successive fractions, respectively. The fractions containing the isolated glycols are made up to 100 ml with H_2O, and the concn. of glycol in an aliquot of each is determined by oxidation with 0·1 *N* $Na_2Cr_2O_7$ in conc. H_2SO_4, followed by measurement of the extinction of the soln. at 610 mμ against a blank (*cf. Anal. Abstr.*, 1956, **3**, 3371). The accuracy is satisfactory for mixtures (25 to 60 mg) containing from 1 to 17 mg of each glycol. W. J. Baker

2233. **Quantitative separation and determination of glycol mixtures by azeotropic distillation.** H. M. Rosenberger and C. J. Shoemaker (A. B. Dick Co., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 100–102.—Three-component mixtures of water, ethanediol and glycerol are quant. separated by azeotropic distillation involving the use of three entrainers. Benzene entrains only water; tetrachloroethylene forms azeotropes with both water and ethanediol but not glycerol; (+)-limonene carries over all three components. Analysis of synthetic mixtures containing between $\simeq 1$ and $\simeq 80\%$ of the three components gives relative errors generally within $\pm 1{\cdot}0\%$. G. P. Cook

2234. **Cerate - chromate oxidimetry. I. Oxidation of carbohydrates.** N. N. Sharma (Univ. of Allahabad, India). *Z. anal. Chem.*, 1957, **154** (5), 340–344 (in English).—In earlier work (*cf. Anal. Abstr.*, 1956, **3**, 3089) ceric sulphate was used in the presence of dil. H_2SO_4 for the oxidation of aldehydic sugars to the formic acid stage only, whereas ketonic sugars were oxidised to CO_2 and H_2O. It is now shown that formic acid itself can be oxidised by refluxing with $Ce(SO_4)_2$ in the presence of Cr^{3+} [1% (w/v) chrome alum] and conc. H_2SO_4. Preliminary work indicates that the Cr^{3+} are initially oxidised to CrO_4^{2+}, which in turn oxidise the formic acid. This new reagent thus combines the oxidising power of both $Ce(SO_4)_2$ and chromate soln., and like pure $Ce(SO_4)_2$ exhibits great stability even on prolonged boiling. Experimental details are given for the determination of glucose, fructose, sucrose, lactose and xylose and results show excellent recoveries in the range 2 to 6 mg. As the earlier method distinguished between aldehydic and ketonic sugars, whereas in the present method both types are completely oxidised to CO_2 and H_2O, it is suggested that a combination of the two methods can be used for determination of mixtures of these two types of sugar.
D. F. Phillips

2235. **Rapid chromatographic method for sugars using glass paper impregnated with silicic acid.** J. W. Dieckert and N. J. Morris (U.S. Dept. Agric., New Orleans, La., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 31–32.—Sucrose, raffinose, D-glucose, L-fructose, D-xylose and L-rhamnose were successfully separated. The developing solvent was a mixture of ethyl ether, phenol, acetone and H_2O, and the spray reagents used for locating the sugars on the chromatogram were conc. H_2SO_4 or *p*-anisidine phosphate. G. P. Cook

2236. **Spectra and spectrophotometric analysis of chlorinated organic compounds. I. Determination of chlorex [di-(2-chloroethyl) ether] in mixtures with dichloroethane.** Ts. N. Roginskaya and A. I.

Finkel'shteĭn. *Zhur. Anal. Khim.*, 1956, **11** (5), 602–605.—In the u.v., soln. of di-(2-chloroethyl) ether (**I**) in dichloroethane (**II**) show a max. absorption at 267·5 mμ but **II** shows no absorption. This can be used to determine **I**, but the method is unsuitable for the analysis of samples containing resin and rubber. In the i.r., an absorption band at 8·50 μ can be used for determining **I** in the presence of **II**, resins and dissolved rubber. The method is sensitive to 0·3% and the error is ±5·5% of the content. Raman spectra excited by Hg 4358 A can also be used for determining **I** in similar mixtures. G. S. SMITH

2237. **Ethyl acetate.** British Standards Inst. (2, Park St., London). B.S. 553:1956, 9 pp.—The specification includes standards and methods of determination for specific gravity at 15·5°/15·5°, 20°/20°, and 25°/25°; distillation range; residue on evaporation; H_2O content; acidity; and ester content. O. M. WHITTON

2238. ***n*-Butyl acetate.** British Standards Inst. (2 Park St., London). B.S. 551: 1956, 9 pp.—The specification includes standards and methods of determination for specific gravity at 15·5°/15·5°, 20°/20°, and 25°/25°; distillation range; residue on evaporation; H_2O content; acidity; and ester content. O. M. WHITTON

2239. **Polarographic determination of vinyl acetate.** Shiro Usami (Toyama Plant, Kurashiki Rayon Co. Ltd., Toyama). *Japan Analyst*, 1956, **5** (9), 499–502.—The polarographic reduction of vinyl acetate (**I**) was studied in methanol containing Hg acetate (0·001 *M*), $NaNO_3$ (0·1 *M*) and thymol blue (0·001 *M*, max. suppressor). An addition compound of **I** and Hg shows a reduction wave at −0·35 V *vs.* the S.C.E., which is proportional to concn. of **I** < 0·001 *M*. This wave is not affected by impurities in **I**, including acetic acid, acetaldehyde and crotonaldehyde. The wave height remains unchanged for 2 hr. Styrene, vinyl butyrate and allyl alcohol are determined similarly. K. SAITO

2240. **Studies with hydroxamic acid. VII. Specific detection of formic esters, their investigation in mixtures, particularly with other carboxylic esters.** O. A. Guagnini and E. E. Vonesch (Nat. Univ., La Plata, Argentina). *Mikrochim. Acta*, 1956, (12), 1767–1773 (in Spanish).—The formic esters are converted into formohydroxamic esters by treatment with hydroxylamine in an aq. alcoholic ammoniacal medium, and detected from the red - violet coloration given with $Fe(ClO_4)_3$. Under the specified conditions, other carboxylic esters, and various other compounds, *e.g.*, carboxylic acids, amides, nitriles and acid chlorides, do not interfere, but imides introduce an error. The method is sensitive to 5 μg or 1 p.p.m. of the formyl radical; other esters (× 10,000) cause no interference. L. A. O'NEILL

2241. **Determination of aldehyde in acetic acid.** Yuzi Takayama (Mitsubishi Rayon Co., Kyobashi, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (9), 1021–1024.—The rate of decay of the violet colour produced by the aldehyde in commercial acetic acid on the addition of Schiff reagent (**I**) was studied at pH 2·8 and 0·7, and, contrary to usual belief, it was found that the aldehyde is formaldehyde (**II**). The determination of a small amount of **II** was studied with **I** and chromotropic acid; both provide satisfactory results. The sample (3 ml) is diluted to 20 ml with **I** (0·2 g of rosaniline hydrochloride, 2 g of $NaHSO_3$ and 2 ml of conc. HCl in 200 ml; pH 2·8; decolorised with activated charcoal) (2 ml), and the extinction is measured at 570 mμ after 70 min. The working curve is linear for < 5 mg of **II** in 100 ml of the final soln. A similar method is applicable to the analysis of acetic anhydride. K. SAITO

2242. **The polarographic determination of small quantities of acraldehyde in the presence of saturated aldehydes.** V. P. Granenova and V. A. Molodovskiĭ. *Trudy Gor'kovsk. Politekhn. Inst.*, 1955, **11** (13), 13–16; *Ref. Zhur., Khim.*, 1956, Abstr. No. 51,202.—Introduce 25 ml of 0·1 *N* NaCl and 25 ml of 0·1 *N* HCl into a cell and pass CO_2 for 10 min. Keep at 25° for 20 min. and take a polarogram of the background; then introduce a freshly prepared soln. containing acraldehyde and take another polarogram. The height of the waves on the reduction curves less that of the residual current is measured. The mean relative error is 5·5%, which is decreased with increase of concn. of the acraldehyde. The presence of saturated aldehydes does not cause interference in an acid medium. C. D. KOPKIN

2243. **Photometric determination of acetone and some other methyl ketones.** V. Šedivec (Inst. Hyg. and Ind. Diseases, Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (1), 63–67.—When treated with an alkaline soln. of hypobromite (10 ml of 40% NaOH soln. added to 100 ml of satd. bromine water), acetone is converted into bromoform, which yields with pyridine a red colour suitable for photometric purposes. *Procedure*—Measure hypobromite soln. (5 ml) into a volumetric flask (25 ml), add an aq. soln. (5 ml) of the methyl ketone to be determined, mix well and set aside for 5 to 10 min. Remove the unchanged hypobromite by adding a soln. of $Na_2S_2O_3$ (10%) (1 ml), then add pyridine (10 ml). Mix well, close the flask and immerse it in a water bath (50°) for 18 to 20 min. Cool with ice, dilute to vol. and measure the intensity, with a green filter. Make a blank determination. Compare the results with a calibration curve, prepared from pure acetone (0 to 250 μg) under the same conditions. Ethyl methyl ketone, methyl propyl ketone and acetylacetone treated in the same manner do not react quantitatively. In the determination of acetone, formaldehyde, methanol, *n*-propanol, *n*-butanol, *iso*butyl alcohol, *iso*amyl alcohol and a fourfold excess of ethanol do not interfere. Acetaldehyde, *iso*propyl alcohol, *sec.*-butyl alcohol, lactic acid and other compounds possessing CH_3CO– and $CH_3CH(OH)$– groups must be absent. J. ZÝKA

2244. **Determination of free acetone in acetone cyanohydrin with hydroxylamine hydrochloride.** Hiroo Sasuga (Inst. Tech.-anal. Chem., Fac. Engng, Tokyo Univ., Hongo, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (10), 1117–1119.—Acetone in acetone cyanohydrin reacts with hydroxylamine hydrochloride (**I**) to give free HCl, which can be titrated with 0·1 *N* Na_2CO_3. At the end-point the pH is 3·5; a mixture of methylene blue and dimethyl yellow (both 0·1% ethanolic soln.) (3 + 5) is the best indicator. Aq. **I** soln. (0·2%) is neutralised with 0·1 *N* Na_2CO_3 to the indicator and the sample (2 to 3 g) is added to this soln. The standard soln. is gradually added until the colour remains unchanged for 1 min. The presence of HCN does not vitiate thee stimation. The titration can also be carried out with the aid of a pH meter. K. SAITO

2245. **Determination of organic substances by standard chromous chloride solution. Nitroso compounds.** N. H. Furman and R. S. Bottei (Princeton Univ., Princeton, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 121–122.—The method is based on the reduction of nitroso compounds by $CrCl_2$ soln., the excess of which is determined potentiometrically with ferric alum soln. Application to the determination of dimethyl-*p*-nitrosoaniline, the ammonium salt of N-nitrosophenylhydroxylamine (cupferron), N-nitrosodiphenylamine and N-benzyl-N-nitrosoaniline resulted in errors of generally less than ±2·5%. G. P. Cook

2246. **Micro-determination of primary amines. I. An improved apparatus for the Van Slyke method.** Daizo Shimoe (Kurosaki Factory, Mitsubishi Chem. Ind. Ltd., Yawata, Fukuoka Prefecture). *Japan Analyst*, 1956, **5** (9), 518–521.—Apparatus for the Van Slyke method was modified with reference to the absorption of NO. To drive off the last trace of NO from the soln., the use of CO_2 (from solid CO_2) is recommended. The use of a mixture of satd. $KBrO_3$ soln. (36 ml) and H_2SO_4 (1 + 1, 12 ml) appears to make for smooth absorption, especially when heat is applied to aromatic amines, which brings about vigorous evolution of NO. The effect of heating time, rate of CO_2 flow and the amount of 40% Na_2SO_3 on the determination of sulphanilic acid was studied and it was found that the improved method increases the yield under these conditions.

II. Abnormal reaction in the Van Slyke method. Daizo Shimoe. *Ibid.*, 1956, **5** (10), 547–550.—An abnormal reaction in the Van Slyke method, resulting from the formation of oximes from active methylene groups (acetoacetic acid, ascorbic acid, etc.), or from the nitrosation of phenols, cannot be suppressed by the addition of KI, especially when heating has to be applied. Such an abnormal reaction appears to arise from an excess of $NaNO_2$ (20%, 0·5 ml), which promotes nitrosation or oxime formation and gives high values of N. When the time of heating (to decompose the diazonium compound) is prolonged, decomposition of oxime is promoted, which also results in high values for N. The amount of $NaNO_2$ must therefore be decreased (1% $NaNO_2$, <1·4 ml) to less than four times the equiv. of the amine.

III. Determination of aromatic amines. Daizo Shimoe. *Ibid.*, 1956, **5** (10), 550–554.—Since the abnormal reaction is inhibited by the use of a smaller amount of $NaNO_2$ (see above), primary amines can be satisfactorily determined by a slightly modified Van Slyke method. The use of KBr (3 *M*, 2 ml) and $CuCl_2$ (4 *M*, 1 ml) is recommended for promotion of diazotisation and decomposition of the diazonium compound, respectively. The amount of $NaNO_2$ is decreased to less than twice the equiv. of the amine. A glycerol bath (145°) is used for heating. This method is better than the diazo titration method, especially in the presence of hydroxy groups, the relative error being ±1·0%. K. Saito

2247. **Photometric determination of aliphatic amines.** H. M. Hershenson and D. N. Hume (M.I.T., Cambridge, Mass., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 16–19.—The addition of aliphatic amines and certain other basic substances to an ethanolic soln. containing excess of $CuCl_2$ produces a colour change from green to yellow which is measured at 860 mμ. The method can be applied directly to ethanolic soln. of aliphatic amines; in the presence of interfering basic substances and water the amines must first be extracted into $CHCl_3$. The concn. range is 10 to 100 μmoles per ml for the extraction procedure and 0·5 to 3·0 μmoles per ml for the direct determination. The standard deviation for the direct procedure is ±1·9% for 11 determinations at the 25-μmole level, and the standard deviation for the extraction procedure is ±3·6% for 14 determinations at the 20-μmole level. The mean recovery by the extraction procedure is 72·9%. G. P. Cook

2248. **Potentiometric titration of hydroperoxides and per-acids in anhydrous ethylenediamine.** A. J. Martin (E. I. du Pont de Nemours & Co., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 79–81.—Per-acids, primary and secondary hydroperoxides, and H_2O_2 can be titrated potentiometrically as weak acids with 0·25 *N* Na aminoethoxide in anhyd. ethylenediamine in a 200-ml titration cell with an antimony indicator electrode. The curves can be interpreted qual. and quant., the potential (0·54 to 0·83 V) at half-neutralisation being related to the acid strength (pK_α = 8·2 to 12·1) and peroxide structure. The error is ≃2%, and, provided that the acidities differ enough, the method is applicable to mixtures, *e.g.*, of H_2O_2 and ethyl hydroperoxide; the results compare well with those obtained by polarography. Interference arises from any substance that masks the peroxide inflection. W. J. Baker

2249. **Potentiometric determination of Complexone III with mercuric salt.** J. Polčin (Res. Inst. Cellulose, Bratislava, Czechoslovakia). *Chem. Listy*, 1957, **51** (1), 168–170.—The determination is carried out in aq. NH_3 soln., with a mercury indicator electrode. The method can be used for the indirect titrimetric determination of Ca, Mg, Cd, Hg, Pb and Cu, when a known vol. of EDTA (disodium salt) (**I**) is added and the excess is determined by mercurimetric titration. *Procedure*—Add a known excess of 0·1 *M* **I** to a neutral soln. of the sample, add buffer soln. (aq. NH_3 - NH_4Cl) (20 ml), dilute to 50 ml and add sufficient Hg to form a 0·5-cm layer in the titration vessel. Immerse a platinum wire into the Hg, connect with a mercurous chloride electrode and adjust the potential to between 160 and 200 mV by adding the buffer soln. or acetic acid. Titrate with 0·1 *M* Hg $(NO_3)_2$. Iron can be masked with tartaric acid or with ammonium tartrate. J. Zýka

2250. **Determination of acrylonitrile [vinyl cyanide] by means of sodium sulphite.** A. P. Terent'ev and S. I. Obtemperanskaya (M.V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1956, **11** (5), 638–639.—The method is based on the determination of the amount of NaOH produced in the reaction—

$$CH_2{:}CHCN + Na_2SO_3 + H_2O = CH_2(SO_3Na)CH_2CN + NaOH.$$

The sample of vinyl cyanide (0·05 to 0·08 g) is contained in an ampoule which is broken under 3 to 5 ml of dioxan, 25 ml of 0·25 *N* Na_2SO_3 is added and the NaOH formed is titrated after 1 to 1·5 hr. with 0·1 *N* HCl in the presence of thymolphthalein. The method is accurate to ±0·2%. G. S. Smith

2251. **Colorimetric estimation of ligands through the effect of their cobalt (III) complexes on redox indicators.** J. S. Hanker, I. Master, L. E. Mattison and B. Witten (Chem. Warfare Lab., Army Chem. Center, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 82–84.—Co-ordination of Co^{3+} with certain ligands,

e.g., dithioethers or amines, increases the ease of oxidation of Co^{2+} to Co^{3+}, the resulting complexes having typical absorption spectra. Because Co^{3+} are easily reduced, some of these complexes act as oxidising agents towards such redox indicators as benzidine, *o*-dianisidine and *o*-tolidine. Microgram amounts of ligand present can thus be determined colorimetrically by utilising either the visible or u.v. spectrum of the complex. Procedures for certain ligands in either aq. or ether soln. are given. A dithioether, *e.g.*, 1:2-di(methylthio)ethane, can be determined in the presence of a monothioether or alkyl sulphide, provided that a few drops of 3% H_2O_2 are present in the soln. when preparing the complex. W. J. BAKER

2252. **Identification of dialkyl sulphides as dialkyl *p*-bromophenacylsulphonium picrates.** S. Veibel and B. J. Nielsen (Dept. Org. Chem., Univ. Technol., Copenhagen, Denmark). *Acta Chem. Scand.*, 1956, **10** (9), 1488–1490 (in English).—Dialkyl sulphides, but not alkyl aryl or diaryl sulphides, react with *p*-bromophenacyl bromide to give the corresponding sulphonium bromides (*Anal. Abstr.*, 1955, **2**, 1251). With diallyl sulphide or dibenzyl sulphide, the product is di-(*p*-bromophenacyl) sulphide. The dialkyl *p*-bromophenacylsulphonium picrates can be quant. titrated with $HClO_4$ in glacial acetic acid, with crystal violet as indicator. A. R. ROGERS

2253. **Determination of organic disulphides by reduction with sodium borohydride.** C. R. Stahl and S. Siggia (Gen. Aniline and Film Corp., Easton, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 154–155.—Organic disulphides are determined by reduction to the thiol with sodium borohydride and aluminium chloride. After reduction, the excess of reductant is destroyed by treatment with NaOH followed by HNO_3 and the thiol is determined potentiometrically with standard $AgNO_3$. The precision and accuracy are within ±1%. Sulphides do not interfere and interference from thiols is corrected for by titration of an unreduced sample. G. P. COOK

2254. **Partition of methylchlorosilanes by gas-liquid chromatography.** K. Friedrich (Res. Inst. Org. Syntheses, Pardubice-Rybitví, Czechoslovakia). *Chem. & Ind.*, 1957, (2), 47.—Six silanes (including tetrachlorosilane and trimethylchlorosilane) were separated with nitrobenzene on infusorial earth as the stationary phase. The gas stream was passed into 0·02 *N* KCl; changes in acidity are determined by conductivity measurements. N. E.

2255. **Infra-red spectrophotometric determination of impurities occurring in α-chlorotoluene (benzyl chloride).** S. K. Freeman (Benzol Products Co., Newark, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 63–68.—A method for the determination of *o*- and *p*-chlorotoluene, *o*- and *p*-chloro-α-chlorotoluene and αα-dichlorotoluene (benzylidene chloride) in benzyl chloride is described. The absorption measurements are made in the 9·0 to 12·2-μ region and the concn. range of the method is between 1 and 10%. A differential procedure enables *o*-chlorotoluene and αα-dichlorotoluene concn. of as low as 0·02%, and *p*-chlorotoluene concn. down to 0·01%, to be determined. The i.r. spectra of the various compounds are illustrated. G. P. COOK

2256. **Quantitative bromination of phenols.** B. Smith (Inst. Org. Kemi, Chalmers Tekniska Högskola, Göteborg, Sweden). *Acta Chem. Scand.*, 1956, **10** (10), 1589–1596 (in English).—The development of the "direct" and "excess" bromination methods of estimating phenols is reviewed and the best conditions for the two methods are presented. *Direct method*—Dissolve 1·5 milli-equiv. of the phenol in a prescribed vol. of glacial acetic acid, usually 10 ml. Add 5 ml of conc. HCl, then 16 ml of 0·1 *N* KBr - $KBrO_3$ dropwise with stirring. Add 5 ml of KI soln. (10%) and immediately titrate with 0·1 *N* $Na_2S_2O_3$, with starch soln. as indicator. *Excess method*—Dissolve 1·5 milli-equiv. of the phenol in 10 ml of glacial acetic acid in a stoppered flask. Add 30 ml of 0·1 *N* KBr - $KBrO_3$ soln. and 5 ml of conc. HCl, stopper the flask immediately and shake. Allow to stand for 3 to 4 min., then add 10 ml of KI soln. (10%) and titrate as above. The average errors of the two methods, as between the calculated and determined equiv. wt., are quoted as 0·3% for the direct and 0·4% for the excess method. Results for 47 phenols are given. The behaviour of various types of phenol towards the test conditions is discussed. G. S. ROBERTS

2257. **Colorimetric estimation of 1-naphthol by sodium cupribromide method.** S. Sass, J. J. Kaufman and J. Kiernan (Army Chemical Center, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 143–144.—1-Naphthol reacts with sodium cupribromide to form a blue colour, which is measured spectrophotometrically at 510 mμ. Other phenolic compounds and 2-naphthol do not interfere. The method was applied successfully to napalm mixtures, recoveries being >98·7%. The concn. range is 10 to 50 μg per ml and the method can be made sensitive to 1 μg per ml. G. P. COOK

2258. **Determination of *p*-dihydric phenols by means of potassium ferricyanide (semi-micro method).** V. Ya. Mikhkel'son. *Trudy Tallin. Politekh. Inst.*, 1955, **A** (63), 127–133; *Ref. Zhur., Khim.*, 1956, Abstr. No. 54,849.—The sample (5 to 25 mg) is mixed with 5 to 8 ml of ethanol (if it does not dissolve easily in water), 20 ml of water, 5 ml of a buffer soln. (a soln. of 40 g of $ZnSO_4.7H_2O$ in 80 ml of water is mixed with a soln. of 30 g of hydrated Na acetate in 80 ml of water, and 4 ml of glacial acetic acid is added) and 10 ml of 0·1 *N* $K_3Fe(CN)_6$ (33 g of the salt and 0·2 g of Na_2CO_3 are dissolved in 1 litre of water). The soln. is stirred for 5 min., 2 ml of 15% KI soln. and 5 ml of 5 *N* H_2SO_4 are added, and the liberated iodine is titrated after 2 min. with 0·05 *N* $Na_2S_2O_3$. The method is applicable to the determination of quinol and its homologues (some require a different amount of oxidant). G. S. SMITH

2259. **Reductimetric determination of anthraquinone with vanadium (II) sulphate.** M. Matrka and Z. Ságner (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1957, **51** (1), 68–71.—*Procedure*—Dissolve the sample (≡ 0·0005 mole of anthraquinone) with warming (50°) in acetic acid (70 ml), dilute with water (10 ml) and titrate with 0·1 *N* VSO_4, potentiometrically. The temp. must not fall below 50°, and the titration must be carried out in an oxygen-free atm. Vanadous sulphate can be prepared by a simple reduction of NH_4VO_3 with Zn in H_2SO_4 medium; its soln. must be stored in the dark and under an atm. of N, and can be standardised by titration with Fe^{3+}. The method has been found suitable for the control of technical samples of anthraquinone. The results were in good agreement with those from polarographic determination.

J. ZÝKA

2260. **Determination of organic substances by standard chromous chloride solution. Diazonium compounds.** R. S. Bottei and N. H. Furman (Princeton Univ., Princeton, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 119–120.—Some diazonium compounds are quant. reduced to the corresponding hydrazine derivative by treatment in neutral or 0·05 *N* HCl medium with an excess of $CrCl_2$, which is determined by potentiometric titration with ferric alum soln. Errors, generally within ±1·5%, were obtained when the method was applied to *p*-ethylbenzylamino-, *p*-diethylamino- and *p*-ethylhydroxyethylamino-benzenediazonium chlorides.

G. P. Cook

2261. **Estimation of arylamides of 3-hydroxy-2-naphthoic acid and related compounds.** E. H. Daruwalla and N. F. Desai (Univ. Bombay, Bombay, India). *Anal. Chem.*, 1957, **29** (1), 116–118.—An alkaline soln. of the arylamide of 3-hydroxy-2-naphthoic acid is titrated with HCl in aq. medium, with phenolphthalein as indicator, and in an excess of ethanol with Alkali blue 6B as indicator. The first titre corresponds to the total amount of alkali present and the second to the free alkali concn. Good agreement was reached with results from a gravimetric and a u.v. absorption method.

G. P. Cook

2262. **Amperometric titration of dodecylpyridinium bromide with sodium dodecyl sulphate.** Nobuyuki Tanaka and Reita Tamamushi (Dept. of Chem., Fac. of Sci., Tohoku Univ., Sendai). *Nature*, 1957, **179**, 311–312.—Dodecylpyridinium bromide soln. (0·1 to 1·0 m*M*) (**I**) were titrated amperometrically at −1·325 V *vs.* the S.C.E. with Na dodecyl sulphate soln. (2 or 10 m*M*) (**II**) in 0·1 *M* NaOH - 0·2 m*M* polyoxyethylene dodecyl ether, with a dropping-mercury electrode. In each titration, 20 ml of **I** was run into a titration cell and the dissolved O was removed by passing purified N through the soln. The current was measured nearly 6 min. after each addition of the titrating agent. All the curves were of the typical *L*-type, from which the equiv. points were determined. With a large excess of **II**, the ppt. formed began to dissolve, and small polarographic waves appeared at −0·9 and −1·2 V *vs.* the S.C.E., but this did not interfere with the titration process. The titration curves gave very sharp end-points, and the standard deviation was ±0·005 m*M* in the concn. range 0·1 to 1·0 m*M* **I**.

W. J. Wright

2263. **Analysis of tertiary amines in xylidine fraction by use of infra-red spectra. II. Detection and determination of 2:3-*cyclo*pentenopyridine.** Shigeyuki Tanaka and Hisakazu Arakawa (Inst. Techno-anal. Chem., Fac. of Engng, Tokyo Univ., Hongo, Tokyo). *Japan Analyst*, 1956, **5** (9), 513–515.—An unknown substance having max. i.r. absorption at 12·8 μ, obtained in the fractionation of the xylidine fraction of coal tar (*Anal. Abstr.*, 1956, **3**, 3103), was identified as 2:3-*cyclo*pentenopyridine. Its max. absorption at 12·76 μ coincides with a characteristic absorption of quinoline, but it is determined with a standard deviation of 1% by the use of two key bands at 12·76 and 12·46 μ (another absorption band of quinoline).

K. Saito

2264. **Vanadatometric determination of 5:7-dibromo[-8-]hydroxyquinoline and dibromohydroxyquinolinates.** V. A. Nazarenko and S. Ya. Vinkovetskaya. *Zhur. Anal. Khim.*, 1956, **11** (5), 572–577.—In the reaction between NH_4VO_3 and 5:7-dibromo-8-hydroxyquinoline (**I**), 10 g-equiv. of NH_4VO_3 are used and pyridine-2:3-dicarboxylic acid and formic acid are produced. To determine **I**, its soln. in 75 ml of 10 *N* H_2SO_4 is boiled for 1 min. with 25 to 50 ml of 0·05 *N* NH_4VO_3, then left on a boiling-water bath for 1 hr., cooled and titrated with 0·05 *N* Fe^{2+}. The amount of **I** that is taken should be such as to leave an excess of +5 to 6 ml of 0·05 *N* NH_4VO_3. To determine Cu, Fe, Ga, Ti and Mo, their compounds with **I** are pptd. and separated, then dissolved in 10 *N* H_2SO_4 and treated with excess of 0·05 *N* NH_4VO_3, etc. Germanium and Si are determined by first forming the molybdo-germanic and -silicic acids, which can then form ppt. with **I**.

G. S. Smith

2265. **Simple determination of 4-amino-5-aminomethyl-2-methylpyrimidine dihydrochloride.** Kimio Uematsu (Sankyo Co. Ltd., Takamine Lab., Shinagawa-ku, Tokyo). *Ann. Rep. Takamine Lab.*, 1955, **7**, 132–135.—The gravimetric determination of 4-amino-5-aminomethyl-2-methylpyrimidine dihydrochloride (**I**) (an intermediate in the prep. of thiamine) was studied in the presence of 4-amino-5-hydroxy-2-methylpyrimidine, 4-amino-5-formyl-2-methylpyrimidine and NH_4Cl. **I** produces colourless needles when boiled with an equimolar amount of urea in a neutral aq. soln., which are sol. in HCl. The sample (≃2 g) is boiled under reflux with urea (3·5 g) and water (20 ml) for 1·5 hr. The product is treated with conc. HCl (2 ml) and filtered, and the filtrate is neutralised with 30% NaOH to pH 9·0; the ppt. is kept in an ice-box for 2 hr., filtered off, washed with cold water (100 ml) and then with cold methanol (10 ml), and dried at 100° to 120° for 2 hr.

K. Saito

2266. **Determination of 4-amino-5-aminomethyl-2-methylpyrimidine.** Satoshi Mizukami, Tameyuki Amano and Shigeko Sakamoto (Res. Lab. Shionogi & Co. Ltd., Imafuku, Amagasaki, Hyogo Prefecture). *J. Pharm. Soc. Japan*, 1956, **76** (9), 1082–1084.—Pifer's ninhydrin method for the determination of 4-amino-5-aminomethyl-2-methylpyrimidine (**I**), an intermediate in the prep. of thiamine (*J. Amer. Pharm. Ass.*, 1951, **40**, 612), was modified for application to the analysis of reaction mixtures. Ammonia and ammonium salts must be removed before the addition of ninhydrin. No interference results from the presence of Na acetate, NaCl and 5-acetamidomethyl-4-amino-2-methylpyrimidine. The sample soln. containing 50 to 300 μg of **I** is made pH 4 with phosphate - citrate buffer and a portion (≃1 ml) is treated with 50% pyridine (1 ml) and 1% ninhydrin in a boiling-water bath for 30 min. The product is cooled, made up to 25 ml and the extinction measured at 570 mμ.

K. Saito

2267. **Identification of solid organic binary systems from the eutectic melting temperature.** M. Kh. Gluzman and V. P. Rubtsova (Kharkov Sci. Res. Chem.-Pharm. Inst.). *Zhur. Anal. Khim.*, 1956, **11** (5), 640–643.—The contact fusion apparatus of Gluzman *et al.* (*Zhur. Prikl. Khim.*, 1953, **26**, 1223) is used in a study of eutectic melting-points of 26 binary organic systems. A method of identifying individual solid organic substances and binary mixtures with any ratio of the components is described.

G. S. Smith

2268. **Separation of the C_{18} to C_{25} fraction of petroleum.** B. J. Mair, W. J. Marculaitis and F. D. Rossini (Carnegie Inst. Technol., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 92–100.—The hydrocarbon types separated and their percentage by vol. in this fraction were—*n*-paraffins (17·1), branched paraffins and *cyclo*paraffins (56·3), mononuclear aromatics (12·2), dinuclear aromatics (7·9), trinuclear aromatics (5·1), residue (1·4). The separation techniques used included adsorption on silica gel and alumina, distillation and crystallisation. Individual *n*-paraffins were also separated.

G. P. Cook

2269. **Determination of copper in fuel oil and other petroleum products.** D. M. Zall, R. E. McMichael and D. W. Fisher (Naval Engng Experimental Station, Annapolis, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 88–90.—The method is based on the colour formed by Cu with neocuproine (2:9-dimethyl-1:10-phenanthroline). The oil sample is dissolved in $CHCl_3$ and the reagent, dissolved in *iso*propyl alcohol soln., is added; the colour, which forms almost instantaneously, is measured at 454 mμ. The results agreed within $\pm 4\%$, over the range of $\simeq 1$ to 105 p.p.m. of Cu, with those from a procedure which involved ashing the sample before the treatment with neocuproine.

G. P. Cook

2270. **A direct infra-red method for determining 2:6-di-*tert.*-butyl-4-methylphenol in mineral oil.** G. H. Bain [Standard Oil Co. (Indiana), Whiting, Ind., U.S.A.]. *Appl. Spectroscopy*, 1956, **10** (4), 193–194.—The method depends on the use of the specific 2·74-μ OH band which is not present in mineral oils. This band is adjacent to a reference extinction characteristic of the phenol-free oil and is free of hydrogen bonding. It is in a range which permits the use of rugged crystal quartz or synthetic sapphire windows. The spectrum of the sample is recorded between 2·65 and 2·85 μ, and the phenol is determined by subtracting the extinction at 2·82 μ from that at 2·74 μ and applying the difference to an appropriate calibration curve. The corresponding phenol-free oil is not needed and no sample preparation is necessary when other substances containing the –OH group are absent. For 14 synthetic samples, each examined once by three analysts, the standard deviation in phenol concn. (0·2 to 0·8%) was 0·006% in precision and 0·01% in accuracy. Twelve samples examined in quintuplicate by five analysts over a period of two months showed a standard deviation of $\pm$ 0·011% of phenol.

K. A. Proctor

2271. **Kjeldahl micro-method for the determination of nitrogen in coal.** M. Dermelj and L. Strauch ("Boris Kidrič" Inst., Ljubljana, Yugoslavia). *Mikrochim. Acta*, 1957, (1), 96–102 (in German).—The Kjeldahl micro-method of Beet (*cf. Anal. Abstr.*, 1954, **1**, 2953; 1955, **2**, 2110) was tested and found to give widely scattered and low results. This is attributed to the use of $KMnO_4$ as catalyst. A method has been devised for amounts of N from 0·1 to 3·0 mg on coal samples of from 5 to 100 mg. The sample is decomposed with conc. H_2SO_4 with the aid of a catalyst mixture [186 g of K_2SO_4, 4 g of $Hg(SO_4)_2$ and 0·8 g of selenium]. The NH_3 is expelled with steam and determined titrimetrically in the usual way. The decomposition time is 4 to 10 min., depending on the type of coal. A reproducibility within $\pm$0·01% is claimed.

D. F. Phillips

2272. **Chromatography of coal-tar dyes.** S. Fujii. *Bull. Nat. Hyg. Lab., Tokyo*, 1955, (73), 335–376.—Data are given for the paper-chromatographic behaviour of 95 artificial coal-tar dyes.

N. E.

2273. **Paper chromatography of [coupling components of the] naphthol AS series and bases [for azo dyes].** Shinya Kitahara and Hachiro Hiyama (Osaka Municipal Ind. Res. Inst., Kitaogimachi, Kita-ku, Osaka). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (10), 1183–1186.—The paper chromatography of 21 commercial developers (coupling components) of the naphthol AS series was studied with various developing agents. The sample (0·01 g) is dissolved in pyridine (2 ml) and one drop is spotted on to filter-paper, developed and revealed with *p*-chlorophenyl diazonium soln. A mixture (1 + 3) of ethanol and aq. NH_3 soln. (5%) is the best developer. The coupling components are satisfactorily identified on the paper by the R_F value and the characteristic coloration. The chromatography of 31 fast bases, such as yellow GC, red BB, orange GR, red GG, etc., was also studied with their *N* HCl soln., by the use of 2% HCl as developer; they are detected by the R_F value and the coloration.

K. Saito

2274. **Paper chromatography of reduction products of naphthol AS dyes.** Shinya Kitahara and Hachiro Hiyama (Osaka Municipal Ind. Res. Inst., Kitaogi-machi, Kita-ku, Osaka). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (10), 1187–1189.—The *o*-methoxyphenylazo dyes of the AS series are reduced with basic $Na_2S_2O_4$ in pyridine and submitted to paper chromatography, with a mixture of butanol, ethanol and water (3 + 1 + 1) as developer. The amino derivatives of the AS series are satisfactorily separated on the paper, the exceptions, including AS–BR, AS–DB, AS–LB and AS–SG, being detected by their characteristic coloration. Cotton pieces (0·5 g) dyed with bases and developers of the AS series are extracted with pyridine (3 ml) and reduced with $Na_2S_2O_4$ at 80° to 90°, and the soln. is submitted to paper chromatography for the detection of the coupling component. For the detection of the diazo component, the cotton piece is treated with $SnCl_2$ in butanol and submitted to paper chromatography, with 2% HCl as developer. The other component does not affect the chromatogram, remaining at the initial point in both cases.

K. Saito

2275. **Chromatography of dyestuffs intermediates. VI. Chromatography of N-arylnaphthylaminesulphonic acids.** J. Latinák and L. Skalický (Východočeské Chem. Závody, Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1957, **51** (1), 91–96.—Conditions have been found for the paper chromatography of N-arylnaphthylaminesulphonic acids, commonly used for the preparation of azo dyes. With the use of this method compounds have been identified which may be present as impurities in azo dyestuff intermediates. The chromatographic behaviour of some dihydroxynaphthalenesulphonic and aminonaphtholsulphonic acids was also studied. The changes of R_F values in the solvent system *n*-propanol - aq. soln. of $NaHCO_3$ (2:1) confirmed the previously derived relations between R_F values and the dissociation of hydroxyl groups (*Anal. Abstr.*, 1957, **4**, 1899). The suitability of the described method (*loc. cit.*)

was tested by analysing a series of technical products. For the detection of the separated compounds, soln. of *p*-nitrophenyldiazonium chloride or *p*-dimethylaminobenzaldehyde were used.
J. ZÝKA

2276. **Determination of ϵ-caprolactam with potassium iodobismuthate.** J. Franc and J. Knížek (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1957, **51** (1), 72–75.—When ϵ-caprolactam (**I**) is treated with a soln. of potassium iodobismuthate, a red compound, $[(C_6H_{11}ON)H]_2BiI_4$, is pptd. and the unchanged iodobismuthate may be determined with a soln. of EDTA (disodium salt) (**II**). The influence of reaction time, temp. and concn. of **I** on the accuracy of the results was studied. *Procedure*—Weigh **I** (0·15 to 1·5 g) into a volumetric flask (100 ml), add water (50 ml) and 0·25 *M* $KBiI_4$ (25 ml) and mix. Add with stirring conc. HCl (10 ml); mix and dilute to vol. with water, and set aside for 3 hr. in a thermostat (20° ± 0·1°), with occasional mixing. Filter through a dry filter-paper. Reject the first 20 ml of the filtrate and to the next 10 ml add buffer soln. (Na acetate - acetic acid) (pH 4·5) (10 ml), 0·1 *N* $Na_2S_2O_3$ (2 ml) and water (10 ml); titrate with 0·05 *M* **II** till the soln. is decolorised; KI soln. (20%) (3 ml) can be added as indicator. Make a calibration curve from 0·1 to 1·8 g of **I**. The average error is ±0·95%. *Reagent*—Dissolve Bi_2O_3 (58·250 g) in conc. HCl (100 ml), evaporate, dissolve the residue in H_2O (500 ml) containing KI (500 g), add conc. HCl (5 ml) and dilute while stirring to 1 litre. Methyl ϵ-caprolactam and **I** in the presence of ϵ-aminocaproic acid can be determined in the same way.
J. ZÝKA

2277. **The determination of the hydroxyl values of organic substances extracted from textile materials.** A. G. Hamlin (British Cotton Ind. Res. Ass., Shirley Inst., Didsbury). *Shirley Inst. Mem.*, 1956, **29** (21), 301–305.—The very simple micro-method devised is based on the Verley and Bolsing procedure (*cf. Ber. dtsch. chem. Ges.*, 1901, **34**, 3354), in which the hydroxyl value is determined by measuring the amount of acetic acid consumed when the sample is acetylated by heating it with a measured vol. of a soln. of acetic anhydride in pyridine. For semi-micro work, 0·1 g of material and 0·4 ml of a soln. of acetic anhydride (1 vol.) in pyridine (3 vol.) were used and for micro work one-tenth of these quantities. Details of procedure and of the simple, easily made apparatus are given. With samples of 0·1 g and 0·01 g, the standard deviations correspond to 1·7 and 3·9 units of acetyl value, respectively.
O. M. WHITTON

2278. **Colorimetric determination of titanium in polyethylene.** R. A. Anduze (Monsanto Chem. Co., Dayton, Ohio). *Anal. Chem.*, 1957, **29** (1), 90–91.—Concn. of Ti as low as 3 p.p.m. in polyethylene can be determined with an error of <2% by the procedure given. The sample (1·5 g) is ignited in a Parr oxygen-bomb; ≯0·3 g of $KHSO_4$ is used to fuse the ashed residue in the prep. of the sample soln. Interfering elements (V, Ni, Cr, Mn, Fe, Mo, Cu and Si) are removed by co-pptn. with zirconium arsenate in 5% H_2SO_4 soln. The mixed arsenates of Ti and Zr are dissolved in 10% H_2SO_4 and, after adding 3% H_2O_2, the extinction of the Ti - peroxide complex is measured at 410 mμ in either a 23- or 50-mm cell. Beer's law is valid for up to 5 mg of Ti. Results are inaccurate if polyethylene is decomposed by acid digestion or by ashing in an open platinum crucible.
W. J. BAKER

2279. **Quantitative determination of metals in poly(vinyl chloride).** E. V. Rouir and M. J. Merkeman (Ateliers de Constructions Electriques de Charleroi, Charleroi, Belgium). *Appl. Spectroscopy*, 1956, **10** (4), 206–209.—The spectrographic determination of Pb (0·004 to 0·1%), Ca (0·0002 to 0·0005%) and Ti (0·01 to 0·2%) is described. The solid sample (2·5 g) is dissolved in ethyl phthalate (100 g), with heating to between 100° and 110° to accelerate solution. Cobalt naphthenate (0·5 g) is added as internal standard and stearic acid (0·1 g) as a spectroscopic buffer. The mixture is then heated at 155° for 60 min. and its spectrum recorded by means of a rotating graphite disc at a speed of 2 r.p.m. The effects of disc speed and temp. have been studied, and the accuracy attainable, as measured by the difference between the average result and theoretical composition of a synthetic mixture, was 3·5% for Pb, 5% for Ca and 2% for Ti.
K. A. PROCTOR

2280. **Determination of residual ammonia in phenol - formaldehyde moulded materials.** Yukio Ieki (Nippon Electric Co. Ltd., Mita-shikoku-cho, Minato-ku, Tokyo). *Japan Analyst*, 1956, **56** (10), 554–556.—Conway's microdiffusion method was applied to the determination of NH_3 (0·002 to 0·3%) in phenol - formaldehyde moulded materials, and the results were compared with those by the A.S.T.M. method. The best indicator is a mixture of bromocresol green (0·033 g) and methyl red (0·066 g) in ethanol (100 ml). The sample (< 150 mesh) (0·5 g) is placed in the outer section of the Conway unit; in the inner section is placed boric acid soln. (10 g of H_3BO_3 dissolved in 200 ml of ethanol and 700 ml of water, neutralised to the mixed indicator and made up to 1 litre) (1 ml). Sodium hydroxide soln. (40%) (2 ml) is added to the outer section and the unit is sealed and set aside overnight. The H_3BO_3 soln. is titrated with 0·02 *N* HCl to a red coloration. The error is 3%.
K. SAITO

2281. **Quantitative determination of amino groups in urea and urea - formaldehyde reaction mixtures of the initial stage, and their chemical structure.** Eisaku Ninagawa (Fac. Educ., Toyama Univ., Gofuku, Toyama). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1956, **59** (10), 1227–1228.—The determination of amino groups in urea and urea - formaldehyde reaction mixtures [containing (hydroxymethyl)urea, di(hydroxymethyl)urea, methylenediurea, (hydroxymethyl)methylenediurea and di(hydroxymethyl)methyleneurea] was studied by the Van Slyke method. The analysis is rapidly carried out by the use of a mixture of acetic acid and H_2SO_4, in place of acetic acid, as solvent. In the decomposition tube, containing acetic acid (2 ml) and 12 *N* H_2SO_4 (0·5 ml), is placed 30% $NaNO_2$ (12 ml); the sample soln. (1 to 2 ml), a small amount of octanol (as foam suppressor) and 12 *N* H_2SO_4 (1 ml) are then added in the order given and shaken for 7 min., and the gas evolved is passed to the nitrometer. The number of amino groups in these addition compounds was also determined, and their possible structure derived.
K. SAITO

2282. **Studies in glycolic titration. III. Titration of organic bases and polyamides in glycol [ethanediol] - phenol medium.** Santi R. Palit and U. N. Singh (Indian Ass. Cult. Sci., Calcutta). *J. Indian Chem. Soc.*, 1956, **33** (7), 507–512.—The use of phenol as a co-solvent in the titration of organic

bases in ethanediol considerably enhances the sharpness of the potentiometric end-point. A visual titration of Nylon against HCl in phenol - ethanediol mixture is carried out with thymol blue as indicator. The value of the mol. wt. obtained by the method is of the right order of magnitude.
I. Jones

2283. **The absorbance ratio method applied to silicone resins.** W. Fishl and I. G. Young (International Resistance Co., Philadelphia, Pa., U.S.A.). *Appl. Spectroscopy*, 1956, **10** (4), 213–215.—The method described permits an i.r. estimation of the relative amounts of phenyl and alkyl groups present in a silicone resin. Owing to the lack of a suitable standard, no determination of absolute concn. can be made. To obtain suitable samples, the resins were spread on flat salt plates and air-dried for a few min.; the procedure was repeated until a check on the bands used (3030 cm^{-1} for phenyl and 2960 cm^{-1} for alkyl) showed absorptions of suitable intensity. The samples were then cured according to the manufacturer's instructions. The spectra were recorded on a Perkin - Elmer Model 12C spectrometer with a sodium chloride prism. The precision estimated for the phenyl to alkyl ratio is no better than ±10%.
K. A. Proctor

2284. **The differentiation of inks by electrochromatophoresis.** C. L. Brown and P. L. Kirk (School of Criminol., Univ. California, Berkeley, U.S.A.). *Mikrochim. Acta*, 1956, (12), 1729–1734 (in English).—The technique previously described (*cf.* Karler *et al.*, *Mikrochim. Acta*, 1956, (11), 1585) was applied to the separation of the constituents of 23 inks, with a voltage of about 350 V, and a buffer prepared by dissolving Na acetate (4 g) and glacial acetic acid (0·5 ml) in 1 litre of water. Barbitone, phosphate or borate buffers may also be used. Better separation than by previously described methods was obtained and all the inks were clearly distinguishable. Several typical chromatograms are shown.
P. S. Stross

2285. **Quantitative determination of rubber auxiliary materials. IV.** O. Lorenz and E. Echte. *Kautsch. u. Gummi*, 1956, **9**, WT300–304.—The quant. determination of mercaptobenzothiazole may be effected by its pptn. as a silver salt or by oxidation to disulphide by means of iodine soln. Zinc benzothiazolyl mercaptide can be determined iodimetrically, either directly or after decomposition by acid. Dibenzothiazolyl disulphide is determined, after reduction with $SnCl_2$ soln., as mercaptobenzothiazole. A method is described for the quant. determination of a mixture of mercaptobenzothiazole, zinc benzothiazolyl mercaptide and dibenzothiazolyl disulphide. The conditions are indicated under which a quant. determination of uncombined S is possible in the presence of mercaptobenzothiazole, its zinc salt, disulphide, and zinc salts of fatty acids. In each case the free S is determined after conversion into thiocyanate by argentimetric conductimetric titration.
Rubber Abstr.

2286. **A rapid, simple and precise determination of nitric nitrogen in nitrates, nitrocelluloses and explosives such as nitroglycerin and penthrite. Application of the 'dead-stop end-point' method.** S. Sandi and G. Flanquart (Lab. des Usines d'Explosifs et Prod. Chim. de Billy-Berclau, P-de-C, France). *Chim. Anal.*, 1957, **39** (1), 20–24.—A semi-automatic electrometric titration method is described which, as compared with other methods, has the advantages of rapidity, simplicity and good precision. The max. variation between results is about 3%.
K. A. Proctor

See also Abstracts 2059, 2076, 2145, 2146, 2155, 2410, 2432, 2443.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

2287. **Determination of blood pH and a new cell for blood pH.** J. T. Murray (Biochem. Unit, Dept. of Path., Christchurch Hosp., New Zealand). *Amer. J. Clin. Path.*, 1956, **26**, 83–89.—A method for determining blood and plasma pH under anaerobic conditions is presented. A new blood pH cell is described for which only 0·5 to 0·6 ml of blood is required. Mercury is used instead of liquid paraffin to ensure anaerobic conditions; it is suitable for rapid routine pH determinations, and the electrodes and cell bodies are interchangeable.
R. S. Tonks

2288. **Nephelometric determination of calcium and magnesium with sodium naphthalhydroxamate.** G. Beck and W. Berli (Univ. Berne, Switzerland). *Mikrochim. Acta*, 1957, (1), 24–29 (in German).—A method is described for the determination of Ca and Mg nephelometrically or photometrically in serum, by pptn. with sodium naphthalhydroxamate in the presence of protein as protective colloid. The method depends on the observation that in undiluted serum both Ca and Mg are pptd. by the reagent, but that in a tenfold dilution only Ca is pptd. The limit of detection is 0·1 μg per ml for Ca and 5·0 μg per ml for Mg.
D. F. Phillips

2289. **Estimation of mercury in urine using di-β-naphthylthiocarbazone.** H. Leach, E. G. Evans and W. R. C. Crimmin (Caernarvon and Anglesey Gen. Hosp., Bangor, Wales). *Clin. Chim. Acta*, 1956, **1** (1), 80–84.—Digest 50 ml of urine with 10 ml of 50% H_2SO_4 and about 0·3 g of $KMnO_4$ under reflux. As the colour fades add further 0·3-g quantities until the colour persists for 5 min. or a total of about 1·5 g has been added. Cool and add 50% aq. hydroxylamine hydrochloride (**I**) soln. until the mixture is colourless, add a further 1 ml of **I** soln. and bring to boiling-point, cool, dilute to about 100 ml with H_2O and extract with 20 ml of reagent soln. If the reagent layer turns bright red, add a further 20 ml of reagent and shake again. Allow the layers to separate, run off the $CHCl_3$ layer, dry with anhyd. Na_2SO_4 if not clear, and read at 515 to 520 mμ. Return the reagent to the funnel and shake with 5 ml of 0·1 *N* $Na_2S_2O_3$ soln. to extract Hg, run off the reagent and read again. Refer the difference in the readings to a calibration curve constructed with 5 to 40 μg of Hg in 100 ml of water containing 5 ml of 50% H_2SO_4 and 1 ml of 50% **I** soln. extracted with 20 ml of reagent, reading the colour before and after reversion and plotting the differences on a graph. Prepare the reagent by dissolving 30 mg of di-β-naphthylthiocarbazone (purified by a method described) in 1 litre of $CHCl_3$.
H. F. W. Kirkpatrick

2290. **Colorimetric determination of small amounts of aluminium with alizarin S in biological and other materials.** W. Oelschläger (Landw. Hochschule, Stuttgart-Hohenheim, Germany). *Z. anal. Chem.*, 1957, **154** (5), 321–329.—In the colorimetric determination of traces of Al with alizarin red S, contamination by Al from glassware was avoided by the use of quartz apparatus. Errors due to reagent aluminium impurity were compensated for by using the same reagents when establishing the calibration curve. Separation of Al from other mineral substances was accomplished rapidly and quant. by means of the acetate method, in which iron phosphate acts as carrier for the Al. On heating, this forms the basic Fe^{III} acetate. The Fe was subsequently separated from the Al by means of NaOH, the sodium aluminate being then acidified with H_2SO_4. After addition of alizarin red S soln. (10 ml, 0·1%), the soln. was neutralised with 2 *N* aq. NH_3 (5 drops in excess) and 5 ml of 5% ammonium phosphate soln. The Al alizarinate was then further stabilised by the addition of 20 ml of a stronger buffer soln. of pH 3·92 (230 g of glacial acetic acid and 100 g of Na acetate in 2 litres of water). Under these conditions the colour intensity remained constant for 2 hr. With substances containing much Fe, *e.g.*, blood, steel, etc., it is only necessary to add ammonium phosphate. The method was tested on a synthetic extract of hay over the range 10 to 110 μg of Al. An average blank of $\simeq$2·8 μg of Al due to reagents was found. (*Cf. Anal. Abstr.*, 1957, **4**, 2124). D. F. PHILLIPS

2291. **Quantitative infra-red spectrography. I. Application to the determination of carbon monoxide.** H. Moureu, P. Chovin, L. Truffert and J. Lebbe (Lab. Municipal de Paris). *Chim. Anal.*, 1957, **39** (1), 3–9.—After a general description of various methods of determining an impurity by i.r. spectrophotometry, a particular application to the measurement of CO in blood is given. This can be completed in 15 min., needs only 1 ml or less of blood, and has a precision at least equal to that of classical methods. K. A. PROCTOR

2292. **An improved type of apparatus for manometric micro-determination of carbon dioxide in small volumes of plasma or serum.** F. Rappaport, F. Eichhorn and M. Nutman (Beilinson Hosp., Rehovot, Israel). *Clin. Chim. Acta*, 1956, **1** (4), 305–310.—The apparatus previously described (*cf. J. Clin. Pathol.*, 1956, **9**, 166) has been improved by replacing the magnetic vibrator with a buzzer armature for shaking the contents of the reaction chamber, and by provision for cleaning without dismantling. H. F. W. KIRKPATRICK

2293. **Direct determination of ^{14}C and ^{35}S in blood.** W. W. Burr and D. S. Wiggans (Texas Univ., Dallas, U.S.A.). *J. Lab. Clin. Med.*, 1956, **48** (6), 907–911.—In the method described, about 0·1 ml of the ^{14}C-labelled sample, diluted if necessary, is absorbed on to a disc of lens tissue placed in an aluminium planchette. After drying, the weight of the sample is found by difference and the radioactivity determined with a windowless gas-flow counter or a thin end-window Geiger - Müller tube. Corrections are applied for background activity and self-absorption. Results agree well with those given by an oxidation procedure requiring pptn. of the ^{14}C as $Ba^{14}CO_3$. For samples containing both ^{14}C and ^{35}S, the activity is determined on each planchette ten times during a 40-day period, and the amounts of the two isotopes are calculated from the observed rate of decay by the methods given. W. H. C. SHAW

2294. **Respiration rate of rat-liver cells at low oxygen concentrations.** [**Determination of oxygen.**] I. S. Longmuir (Inst. of Diseases of Chest, Brompton Rd., London, England). *Biochem. J.*, 1957, **65** (2), 378–382.—A polarographic method is described for the simultaneous determination of the concn. of oxygen in a medium and the respiration rate of a tissue in that medium. It is essentially that described by Longmuir (*Biochem. J.*, 1954, **57**, 81), but with some improvements. J. N. ASHLEY

2295. **The determination of alcohol in blood and breath, and its problems.** D. Monnier (École de Chimie, Geneva, Switzerland). *Chimia*, 1957, **11** (1), 16–21.—Various methods of determining ethanol in blood are briefly reviewed and a fairly complete description is given of the "Breathalyser," an instrument developed in the U.S.A. It determines the ethanol content of breath by photometric measurement of the reduction of dichromate at 45° to 50° by a constant known vol. of breath, recording the percentage of ethanol in blood from the relationship 2100 ml of breath contains the same amount of ethanol as 1 ml of blood. The reading is given in 5 min. with a max. error of ±10%. H. F. W. KIRKPATRICK

2296. **Estimation of Fluothane [halothane] in blood.** R. R. Goodall [Res. Dept., Imperial Chemical (Pharmaceuticals), Blackley, Manchester]. *Brit. J. Pharmacol.*, 1956, **11** (4), 409–410.—Halothane, $CF_3CHBrCl$, a new volatile anaesthetic, is extracted from the blood with light petroleum. Aliquots of the extract are heated in sealed ampoules under pressure with sodium amyloxide, and the bromide formed is estimated nephelometrically as AgBr. An improved method, in which reduction by lithium aluminium hydride is used to form the halide, is being developed. G. F. SOMERS

2297. **Determination of low concentration of chloramphenicol in blood with reference to the minimum measurable concentration.** Motohiro Maruyama (Takamine Lab., Sankyo Co. Ltd., Shinagawa-ku, Tokyo). *Ann. Rep. Takamine Lab.*, 1955, **7**, 158–163.—Glazko's diazo-coupling method for the determination of chloramphenicol (**I**) (*J. Biol. Chem.*, 1950, **183**, 679; *Arch. Biochem.*, 1949, **23**, 411) was modified to lower the limit of determination, mainly by decreasing the vol. of the soln. in which the colorimetric procedure is carried out. The results were statistically analysed and attempts made to express the limit of concn. on a statistical basis. The limit is 1·35 μg per ml when the serum is directly submitted to colorimetry, and 3·73 μg when **I** is extracted from serum with ethyl acetate. K. SAITO

2298. **The change of thiacetazone *in vivo*. II. Colorimetric determination of thiacetazone in urine and its extraction.** Takaichi Arita (Pharm. Inst., Med. Fac., Kyoto Univ., Sakyo-ku, Kyoto). *J. Pharm. Soc. Japan*, 1956, **76** (9), 984–987.—Deacetylated thiacetazone (**I**) is extracted with $CHCl_3$ from a basic soln. and treated with acid *p*-dimethylaminobenzaldehyde (**II**) soln. to yield a yellow colour, which obeys Beer's law for < 2 mg of **I** in 100 ml. Coloured substances in urine are removed by extraction with $CHCl_3$ before the addition of NaOH. The presence of isoniazid, streptomycin

and decomposition products of **I**, including *p*-acetylaminobenzoic acid, *p*-aminobenzoic acid and glucuronic acid thiosemicarbazone, does not cause interference with the colorimetry. Sulphonamides and 4-aminosalicylic acid increase the error. *Procedure*—Diluted urine (1 + 3, 4 ml) is heated with 5 *N* HCl (1 ml) for 30 min., then cooled and shaken with $CHCl_3$ (3 ml) for 5 min. The aq. layer (4 ml) is adjusted to pH 13 with 5 *N* NaOH and shaken with $CHCl_3$ (3 ml) to transfer the deacetylated **I** to the organic layer. A 2-ml portion of the $CHCl_3$ soln. is mixed with ethanolic **II** soln. (2%, 1 ml), 5 *N* HCl (0·2 ml) and ethanol (0·5 ml), and the extinction is measured at 430 mμ. K. Saito

2299. **Method for estimation of free and acetylated sulphonamide or 4-aminosalicylic acid in the urine.** M. Brous. *Amer. J. Clin. Path.*, 1956, **26**, 552.—To 2 ml of urine add 5 ml of 4 *N* HCl and dilute with water to 50 ml. Shake and add another 50 ml of water. To 10 ml of this soln. add 0·3 ml of Ehrlich's aldehyde reagent. A yellow colour appears which is proportional to the amount of free sulphonamide or 4-aminosalicylic acid present, and is measured at 460 mμ. For estimation of total sulphonamide or 4-aminosalicylic acid, heat 10 ml of the acidified, diluted soln. for 1 hr. on a boiling-water bath, then proceed as before. At the dilution employed, Ehrlich's reagent does not react with urobilinogen. R. S. Tonks

2300. **Colorimetric determination of nitrofurazone, nitrofurantoin and furazolidone in plasma.** J. A. Buzard, D. M. Vrablic and M. F. Paul (Norwich Pharm. Co., Norwich, N.Y., U.S.A.). *Antibiot. & Chemother.*, 1956, **6** (12), 702–707.—The method described is based on the formation of 5-nitro-2-furfuraldehyde phenylhydrazone, which is extracted into toluene and determined photometrically. *Procedure*—Place 3 ml of plasma (oxalated, citrated or heparinised) in each of two test-tubes (1·8 cm × 15 cm). Prepare the control sample by adding 5 to 10 mg of $Na_2S_2O_4$ to one tube and shaking occasionally during 15 min. or use nitrofuran-free plasma. To each tube and to standards prepared as described below, add 1 ml of 1·5% aq. phenylhydrazine hydrochloride (prepare fresh daily; refrigerate when not in use) and 1 ml of 5 *N* HCl. Mix and heat for 25 min. at 70°. Cool for 5 min. in cold water, add 5·0 ml of toluene, shake vigorously 25 times and centrifuge. Break the curd-like top layer by gentle stirring and remove the toluene. Recentrifuge if not clear and measure the extinction at 430 mμ against toluene as blank. *Standards*—Dissolve 50 mg of cryst. standard nitrofuran in 50 ml of dimethylformamide and dilute 5 ml to 50 ml with water. Dilute, when required for use, 5 ml to 50 ml with water. Measure 1, 2 and 3 ml (10 to 30 μg of nitrofuran) into test-tubes and dilute when necessary to 3 ml. W. H. C. Shaw

2301. **Convenient apparatus for the xanthhydrol method of determining blood urea.** E. M. Abrahamson (60 Sutton Place South, New York, 22, U.S.A.). *Amer. J. Clin. Path.*, 1956, **26**, 103–106.—A new glass apparatus is described which avoids any transference once the unknown solution is placed in it. The method is a very rapid one. R. S. Tonks

2302. **Prevention of colour fading in the estimation of blood sugar after Folin and Wu.** P. G. Higgins and H. Lehmann (Dept. of Pathol., St. Bartholomew's Hosp., London, England). *Clin. Chim. Acta*, 1956, **1** (2), 178–188.—The molybdophosphoric acid reagent of Folin and Wu is modified by adding 215 ml of H_3PO_4 and 35 ml of H_2O to each 500 ml. For the test, 3 ml of this modified reagent is used and the soln. is diluted to 12·5 ml, giving a final concn. of H_3PO_4 of 13% (v/v). This concn. is usually sufficient to stabilise the colour, but may have to be increased with some batches of reagent. H. F. W. Kirkpatrick

2303. **Use of capillary blood for estimating renal clearance of inulin and glucose excretion by means of an anthrone procedure.** M. B. Handelsman and M. Sass (Veterans Admin. Hosp., Brooklyn, N.Y., U.S.A.). *J. Lab. Clin. Med.*, 1956, **48** (5), 759–768.—Inulin and glucose are determined in 0·2 ml of capillary blood at concn. usually attained in renal function studies by a modification of the anthrone method. An aliquot of a protein-free dilution of the sample is heated with a soln. of anthrone in conc. H_2SO_4 for 10 min. at 56°. The reaction is then stopped by cooling and the colour (mainly due to inulin) is measured at 620 mμ. After further heating at 100° for 12 min. the colour is then measured at 540 mμ; this gives the glucose content plus inulin. The amounts of the two compounds are calculated from the extinctions at the two wavelengths by the methods given. A correction for other anthrone-reactive substances of blood may be applied. W. H. C. Shaw

2304. **Semi-micro method for spectrophotometric determination of oxyhaemoglobin : haemoglobin ratio in blood.** F. Geubelle (Clin. et Policlin. des Malades de l'Enfance, Liège Univ., Belgium). *Clin. Chim. Acta*, 1956, **1** (3), 225–228.—Blood is collected out of contact with air, with a little heparin as anticoagulant. Approx. 1 ml is drawn into a syringe containing about 0·3 ml of 5% saponin soln. and a small glass bead to facilitate mixing and haemolysis; after shaking, a 1-mm cell is filled with the haemolysed blood (rejecting the first few drops) and the extinction is read at 650 mμ and 805 mμ. The percentage of oxyhaemoglobin is given by

$$K_1 (D_{805} - D_{650})/(K_1 - K_2)D_{805},$$

where D = concn. × cell thickness × log I_0/I, and K_1 and K_2 are constants determined with blood containing 100% of oxyhaemoglobin and of haemoglobin, respectively. H. F. W. Kirkpatrick

2305. **The separate determination of direct-reacting bilirubin in urine.** F. A. Gries and G. Gries (Med. Univ. Klinik, Marburg a.d. Lahn, Germany). *Klin. Wochschr.*, 1957, **35** (2), 81–85.—Mix 2 ml of urine with 1·5 ml of 11% $Na_2HPO_4.12H_2O$ soln. and 0·5 ml of 20% $CaCl_2.6H_2O$ soln. Shake well, centrifuge after 30 min. and wash the ppt. with a little 0·2% $CaCl_2$ soln. Dissolve the ppt. in about 1 ml of buffer soln. (1·5 *N* acetic acid - 1·5 *N* Na acetate) (9:1), pH 3·8, and 3 to 5 drops of 25% HCl, and dilute with a further 3 ml of the same buffer. Add 1 ml of freshly prepared diazo reagent and adjust to a pH of 3 to 4·2 with buffer or the Na acetate soln. After 1 hr. add 2 ml of 25% HCl and make up to 10 ml with water. After 30 to 60 min. read the extinction at 560 mμ in a 1-cm cell and calculate the bilirubin concn. from extinction × 4·3 = bilirubin in the urine (mg %). H. F. W. Kirkpatrick

2306. **Spectral absorption of coproporphyrin in relation to the estimation in urine.** H. A. Zondag and E. J. van Kampen (Clin. Chem. Lab., Diakonessenhuis, Gröningen, Holland). *Clin. Chim. Acta*, 1956, **1** (2), 127–134 (in English).—To estimate preformed coproporphyrin (**I**), extract 15 ml of urine (pH 5·0 to 5·5) twice with 15 ml of ether - glacial acetic acid (100:5) breaking any emulsion, if necessary, by adding 1 ml of ethanol. Wash the ether extract with 10 ml of 1% Na acetate soln. and then with 10 ml of H_2O. Extract **I** from the ether with 0·1 *N* HCl (3 × 2 ml), measure the vol. of the acid extract and measure the extinctions at 380, 400·5 and 421 mμ. (Clarify the acid soln. if necessary by centrifuging at high speed.) Calculate the corrected extinction from

$$[2E_{400\cdot5} - (E_{380} + E_{421})]/1\cdot843.$$

An $E_{corr.}$ of 0·741 corresponds to 1 μg of **I** per ml in the acid extract. **I** chromogen is not extracted from the ether by 0·1 *N* HCl and is estimated by allowing the acid-extracted ether to stand in the dark for 48 hr. for complete conversion into **I**, re-extracting with 0·1 *N* HCl and reading the extinctions as before. H. F. W. KIRKPATRICK

2307. **The determination of vitamin C in normal human plasma and erythrocytes.** B. Iggo, J. A. Owen and C. P. Stewart (Dept. of Clin. Chem., Univ. of Edinburgh, Scotland). *Clin. Chim. Acta*, 1956, **1** (2), 167–177.—Various methods of estimating ascorbic acid, dehydroascorbic acid and dioxogulonic acid, based on the reduction of indophenol or the formation of a 2:4-dinitrophenylhydrazine derivative, have been applied to human plasma and erythrocytes. It is concluded that measurement of indophenol reduction, at about pH 4, in protein-free filtrates of plasma, or of CO-saturated erythrocytes, treated with *p*-chloromercuribenzoic acid (Owen and Iggo, *Biochem. J.*, 1956, **62**, 675) gives the most accurate estimate in these materials.
H. F. W. KIRKPATRICK

2308. **Vitamin B_{12} in blood. II. Determination of free vitamin B_{12} in blood.** Junzo Hayashi and Kiichi Iwamoto (Biochem. Lab., Shiga Agric. Coll., Hikone, Shiga Prefecture). *J. Agric. Chem. Soc. Japan*, 1956, **30** (7), 399–401.—The determination of free vitamin B_{12} (**I**) in blood was studied by bio-assay with *Euglena* (*Ibid.*, 1954, **28**, 314). Since combined **I** readily dissociates on heating, the sample is treated with metaphosphoric acid (**II**) to remove proteins and added to the culture through a Seitz filter. The amount of 10% **II** must be increased to 1 ml (for 1 ml of blood) to make the pptn. quantitative. Its inhibiting action on *Euglena* is suppressed by the addition of casein hydrolysate. Standard **I** must be added to the culture in the same way as the sample. Blood (1 ml, containing Na oxalate) is treated with water (12 ml), *N* HCl (1 ml) and 10% **II** (1·0 ml), and centrifuged; the supernatant liquid is adjusted to pH 6·5, diluted and submitted to bio-assay.
K. SAITO

2309. **Paper chromatography of the non-volatile organic acids of human urine.** R. Osteux and J. Laturaze (Lab. de Chim. Biol., Fac. Med. et Pharm., Lille, France). *Clin. Chim. Acta*, 1956, **1** (4), 378–391.—The urine was purified by passage first through Amberlite IR4B and then through Permutit 50. The soln. was chromatographed on paper with an alkaline solvent (ethanol - 20% aq. NH_3 - H_2O) (80:5:15) and then with an acid solvent (*n*-butanol - formic acid - H_2O) (7:3:12). Identification of the spots was made by various methods; in some instances the spots were eluted and chromatographed in other solvents. Thirty-three organic acids (nine unstable) were separated from human urine. H. F. W. KIRKPATRICK

2310. **The keto acids of blood and urine. Analytical study; identification of new hydrazones; clinical applications.** G. Biserte and B. Dassonville (Faculté de Méd. et de Pharm., Lille, France). *Clin. Chim. Acta*, 1956, **1** (1), 49–62.—Paper chromatography of the 2:4-dinitrophenylhydrazones, with the solvent system *iso*amyl alcohol - ethanol - H_2O (75:15:60), showed the following main keto acids in blood: α-oxoglutaric acid (**I**) (R_F 0·08), two isomeric hydrazones of pyruvic acid (**II**) (R_F 0·22 and 0·39), and two hydrazones of α-oxo*iso*hexoic acid (**III**) (R_F 0·53 and 0·68); acetone (R_F 0·92) was also observed. In urine the differences were predominance of **I** over **II**, appreciably more acetone, and only traces of **III**. A method is described for determining **I** and **II** in biological materials, and its clinical applications are indicated. H. F. W. KIRKPATRICK

2311. **Ion-exchange method of determining carnosine, anserine and their precursors in animal tissue.** C. L. Davey (Low Temp. Res. Sta., Cambridge Univ., England). *Nature*, 1957, **179**, 209–210.—Fractionation was carried out by buffered elution from a 50-cm column (0·9 cm diam.) packed with Dowex-50 resin, 4% cross-linked, by using the method of Moore and Stein (*J. Biol. Chem.*, 1951, **192**, 663; 1954, **211**, 893). Complete recovery of the amino acids containing an iminazole nucleus was attained by adding chelating agents to the eluting buffers to prevent interference by heavy metals. The elution was made with 0·2 *M* borate buffer (pH 8·0) containing 0·03% of Na diethyldithiocarbamate (**I**), 0·181 *N* in Na^+, followed by 0·22 *M* Na_2CO_3 containing 0·03% of **I**. The separated amino acids were determined by a ninhydrin method. H. F. W. KIRKPATRICK

2312. **A procedure increasing the specificity of the Jaffé reaction for the determination of creatine and creatinine in urine and plasma.** H. A. Taussky (Russell Sage Inst. of Pathol., Cornell Univ., New York, U.S.A.). *Clin. Chim. Acta*, 1956, **1** (3), 210–224.—Detailed procedures are given for the application of the Jaffé reaction to blood and urine for the determination of creatinine and creatine plus creatinine. H. F. W. KIRKPATRICK

2313. **Determination of creatine phosphate from the amount of creatinine formed.** A. M. Alekseeva (I.P. Pavlov Leningrad Med. Inst.). *Biokhimiya*, 1956, **21** (2), 243–246.—The method described previously (*Brit. Abstr. C*, 1952, 259) is modified. The sample of tissue (0·3 to 0·5 g) powdered in liquid air is extracted, according to one variant of the method, with 10 ml of 5% trichloroacetic acid for 10 to 15 min., and 1 ml of the filtered soln. is mixed with 1 ml of 1·7% $(NH_4)_2MoO_4$ soln. in *N* HCl and set aside for 30 to 40 min. Then 1·2 ml of 10% NaOH soln. and 2 ml of a saturated soln. of picric acid are added and the soln. is diluted after 10 min. to 25 ml and the creatinine is determined colorimetrically. G. S. SMITH

2314. **Determination of hexosamine-containing compounds in biological materials.** B. N. Gladyshev (A.N. Bakh Inst. of Biochem., Acad. Sci., U.S.S.R.,

Moscow). *Biokhimiya*, 1956, **21** (2), 227–230.—The material is twice treated with acetone in the cold during 2 hr., then vacuum-dried and extracted four times with 40-fold amounts of boiling formaldehyde - ethanol (5 + 1). In the presence of large amounts of lipids, the material is first extracted with ether. A sample (100 to 250 mg), vacuum-dried and then brought to const. wt. at 50°, of the extracted material is hydrolysed by boiling under reflux for 6 hr. with 5 to 10 ml of 6 *N* HCl. The filtered hydrolysate is neutralised with Na_2CO_3 and $NaHCO_3$, and again filtered. A mixture (1 + 1) of conc. HCl and 30% trichloroacetic acid (1·25 vol.) is added to 3 to 4 ml (1 vol.) of the neutralised hydrolysate. Solid Na_2CO_3 (0·5 vol. from a graduated cylinder; 1 g of the powder ≡ 1 ml approx.) is added all at once and the soln. is decanted through a filter. The amino-sugar content is determined colorimetrically by the method of Elson and Morgan (*Biochem. J.*, 1933, **19**, 656). G. S. SMITH

2315. **Determination of nucleic acids by their reactions with cysteine and sulphuric acid.** B. Bakay, L. Kirschner and G. Toennies (Lankenau Hosp. Res. Inst., Philadelphia, Pa., U.S.A.). *Arch. Biochem. Biophys.*, 1956, **65** (2), 500–512.—A study is made of the effects of reagent concn. and time and temp. of heating on the colours formed by deoxyribonucleic acid (**I**) and ribonucleic acid (**II**) with cysteine in dilute H_2SO_4. Trichloroacetic acid is recommended for the liberation of the nucleic acids from tissues and tissue proteins, and it also suppresses the interference of **I** in the determination of **II** by measurement of the extinction at 390 mμ. **I** is determined separately by measurement of the extinction at 470 mμ under conditions slightly different from those required for **II**. The contents of both nucleic acids in samples are calculated by the methods given.
W. H. C. SHAW

2316. **Comparison of the merits of the new method of hydrolysis of nucleic acid by phosphoric oxide with those of the standard perchloric acid method.** M. V. Narurkar and M. B. Sahasrabudhe (Indian Cancer Res. Centre, Parel, Bombay, India). *Biochim. Biophys. Acta*, 1957, **23** (2), 367–371.—Ribo- or deoxyribo-nucleic acid is hydrolysed by heating with P_2O_5 at 150° for 2 hr. After cooling, the liberated purine and pyrimidine bases in the charred residue are dissolved in 5 ml of 0·1 *N* HCl and the solution is filtered from carbon particles. The bases are separated by descending chromatography for 20 to 24 hr. on Whatman No. 1 filter-paper, with *iso*propyl alcohol - 5 *N* HCl (68:32) or butanol as developing solvents for hydrolysates of the two respective nucleic acids. Quant. recovery of added bases and of total N (Kjeldahl) in the sample is attained, and results compare well with those obtained by a standard $HClO_4$ procedure.
W. H. C. SHAW

2317. **Interference by reduced pyridine nucleotides in the diazotisation of nitrite.** A. Medina and D. J. D. Nicholas (Long Ashton Res. Sta., Bristol Univ., England). *Biochim. Biophys. Acta*, 1957, **23** (2), 440–442.—Interference by reduced di- or triphosphopyridine nucleotide (DPNH or TPNH) in the determination of NO_3^- or NO_2^- reductase activity by reaction of the NO_2^- with sulphanilamide and N-(1-naphthyl)ethylenediamine hydrochloride is eliminated by the following modification of the method. *Procedure*—At the end of the incubation period add 0·1 ml of *M* barium acetate soln. and 2·5 ml of 95% (v/v) ethanol to the cold reaction mixture. Agitate well and centrifuge at 0°. Complete the determination of NO_2^- on the supernatant liquid.
W. H. C. SHAW

2318. **Chromatography of phospholipids and related compounds on glass paper impregnated with silicic acid.** M. Brown, D. A. Yeadon, L. A. Goldblatt and J. W. Dieckert (U.S. Dept. Agric., New Orleans, La., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 30–31.—Some phospholipids and their hydrolytic cleavage products were separated by chromatography on glass paper impregnated with silicic acid, by using a solvent system composed of phenol, ethyl ether, acetone and water. Choline-containing compounds were detected on the chromatogram with a molybdophosphoric acid - $SnCl_2$ reagent or with a modified Dragendorff reagent (potassium iodobismuthate). Compounds containing a primary amino group were detected by a ninhydrin spray, and substances that char were detected by a conc. H_2SO_4 spray followed by heating. R_F values for 12 compounds are listed.
G. P. COOK

2319. **Electrochromatograms of human bile.** J. C. M. Verschure (Med. Clinics, State Univ., Utrecht, Holland). *Clin. Chim. Acta*, 1956, **1** (1), 38–48 (in English).—Bile (2 to 10 ml) is concentrated in small Cellophane bags by being placed in 15% carboxymethylcellulose soln. in the refrigerator for 48 hr. Paper electrophoresis is then carried out in barbitone buffer (0·06 *M*) (pH 8·6), and after drying at 110° for 15 min. the papers are cut so that 5 to 7 strips per sample are available for staining. Proteins and lipids are stained with azocarmine B and Sudan black, respectively. To stain bilirubin, the strip is sprayed with freshly mixed diazo reagent, prepared by mixing 10 ml of 96% ethanol, 10 ml of 0·1% aq. sulphanilic acid and 0·6 ml of 0·5% aq. $NaNO_2$, and dried in air. Bile acids are stained by spraying with 2% vanillin in 96% ethanol, drying, placing in 85% H_3PO_4 at 70° and heating in an oven at 70° for 4 min., when the bile acids give a cherry-red colour. The paper contracts and disintegrates in the acid, so that a "dummy" strip is made with coloured pencils for comparison with the other strips. Urobilin is detected in u.v. light after spraying with alcoholic Zn acetate and drying. Results with various specimens of bile are illustrated and described.
H. F. W. KIRKPATRICK

2320. **Electrophoretic analysis of bile.** P. Dessi and R. Pellegrini (Inst. of Pharmacol. and Expt. Therapy, Univ. of Bologna, Italy). *Klin. Wochschr.*, 1957, **35** (1), 34–37.—Various specimens of bile were examined by paper-strip electrophoresis, continuous electrophoresis and the Tiselius moving-boundary method. On filter-paper the component bands were observed in u.v. light when, in general, four bands of differing colour fluorescence were observed. The patterns found in dogs' and ox bile and in bile from patients with gallstones and hepatitis are described. H. F. W. KIRKPATRICK

2321. **Electrophoresis of cerebrospinal fluid.** M. Mumenthaler and H. Märki, sen. (Med. Klinik des Kantonsspitals, Winterthur, Switzerland). *Klin. Wochschr.*, 1957, **35** (1), 1–7.—A vol. of 6 to 8 ml of normal fluid, or a smaller vol. of pathological fluid with raised protein content, is centrifuged at 3000 r.p.m. for 10 min. to remove erythrocytes and is then concentrated by ultrafiltration to bring the

protein content to between 6 and 8 % (w/v). For the paper electrophoresis, 0·01 to 0·02 ml of the conc. fluid is used, with a barbitone - acetate buffer of pH 8·6. Results with various fluids and clinical applications are given. H. F. W. KIRKPATRICK

2322. **Paper chromatography of urinary amino acids.** J. Awapara and Y. Sato (Univ. of Texas, Houston, Tex., U.S.A.). *Clin. Chim. Acta*, 1956, **1** (1), 75–79.—Purify the urine by mixing 0·5 to 1 g of activated charcoal with 5 to 10 ml of urine, add 2 to 5 drops of $CHCl_3$ (to prevent adsorption of some amino acids), mix well, warm in a steam bath for 5 min., and filter. To remove salts, concentrate the filtrate to about 1 ml and transfer to a column of Dowex-2 resin (200 to 400 mesh), purified by a method described. Rinse the container with 0·5 ml of H_2O and transfer to the column. Wash the column with cold boiled H_2O until the filtrate is neutral, elute the amino acids with 4 *N* acetic acid, collecting 10 ml of eluate. Evaporate the eluate to dryness and dissolve the residue in 1 ml of H_2O. If the pH of this soln. is not about 4 to 5, repeat the evaporation. Use this soln. for one-dimensional paper chromatography on Whatman 3MM paper with butanol - formic acid - H_2O (75:15:10) as solvent, neutralising the sample by blowing NH_3 vapour on to the area containing the sample. After development and drying, treat with ninhydrin and make a quant. comparison of the areas by any of the usual methods.
H. F. W. KIRKPATRICK

2323. **Quantitative determination of amino acids by paper chromatography. A solvent to replace phenol.** M. Wolfe (Univ. Coll., London, England). *Biochim. Biophys. Acta*, 1957, **23** (1), 186–191.—A one-phase solvent consisting of *n*-butanol - ethyl methyl ketone - 17 *N* aq. NH_3 - water (5:3:1:1, v/v) is recommended in place of phenol as the first developing solvent in the two-dimensional chromatography of amino acids on Whatman 3MM paper. After descending development for 34 hr. at 20°, the solvent may be removed by heating for 3 hr. at 40°. R_F values of 25 naturally occurring amino acids in this solvent are given. *n*-Butanol - acetic acid - water (4:1:5, v/v) is used as the second solvent. Good separations of amino-acid constituents of proteins are attained, and losses during chromatography are shown, by subsequent ninhydrin colorimetric determination, to be $\gtrsim 4\%$.
W. H. C. SHAW

2324. **Determination of amino acids by radio-isotope dilution method combined with paper chromatography.** Tsuru Wada (Inst. of Applied Microbiology, Tokyo Univ., Hongo, Tokyo). *J. Agric. Chem. Soc. Japan*, 1956, **30** (5), 229–232.—The determination ($\simeq 1$ μmole) of alanine, leucine, arginine, glutamic acid, threonine, proline, tyrosine, valine, phenylalanine and *iso*leucine was studied with the aid of ^{14}C-labelled compounds. The sample soln. (0·5 ml) is mixed with the ^{14}C-labelled amino acid to be determined (of known specific radioactivity, 1000 to 2000 counts per min.). One drop is submitted to one-dimensional or two-dimensional paper chromatography with a mixture of *n*-butanol, acetic acid and water (4 + 1 + 1), or of phenol and water (8 + 2) (for two-dimensional development only) by the usual method. The spot, detected by radioautography or with ninhydrin (0·1% in butanol), is cut out, extracted with water (1·5 ml) and the content of amino acid in a 0·5-ml portion is determined colorimetrically by the Moore - Stein method (*J. Biol. Chem.*, 1954, **211**, 907). The specific radioactivity is measured with another 0·5-ml portion, and the amino-acid content calculated. The error is $\simeq 10\%$. K. SAITO

2325. **Determination of amino acids in biological material. III. Chromatographic analysis of basic amino acids on ion-exchange resins.** R. Müller and G. Krampitz (Inst. Anat. Haustiere, Univ. Bonn, Germany). *Z. Tierernähr.*, 1956, **11**, 227–231.—An adaptation of the method of Moore and Stein is described. The length of column preferred is 15 cm and the resin is Dowex 50 X-8 (375 mesh). The reagents for the ninhydrin procedure are 1 g of pure ascorbic acid and 1 g of ninhydrin in 160 ml of 2-methoxyethanol; 20 g of ninhydrin in 1 litre of 2-methoxyethanol; 4 *N* Na acetate buffer. The reagents are mixed in the ratio of 1:2:1 immediately before use, and 1 ml is enough for max. coloration. The samples are well shaken, placed in a water bath for 15 min., and cooled before 15 ml of ethanol and water in equal parts is added, and the mixture is shaken again for 30 sec. The method has been used for bovine serum albumin, skimmed milk powder and barley. NUTR. ABSTR. REV.

2326. **Identification and semi-quantitative determination of phenylthiohydantoins [of amino acids].** P. Edman and J. Sjöquist (Dept. Physiol. Chem., Univ. Lund, Sweden). *Acta Chem. Scand.*, 1956, **10** (9), 1507–1509 (in English).—The method previously described (*Acta Chem. Scand.*, 1953, **7**, 447) for the identification of the 3-phenyl-2-thiohydantoin deriv. of amino acids has been modified to permit a semi-quant. determination based on the u.v. absorption at 269 mμ. Suitable solvent systems for the paper chromatography are *o*-xylene - formamide, *n*-butyl acetate - propionic acid - formamide, and *n*-heptane - 1:2-dichloroethane - 75% aq. formic acid, and R_F values obtained with these systems are tabulated. The position of each deriv. on the paper chromatogram is located with the aid of a mercury lamp and a fluorescent cadmium borate screen. The spot is cut out and eluted with ethanol and the extinction determined in a spectrophotometer. The accuracy of a single determination is ±10%, although the recovery may be as low as 75% for the faster-moving spots. A. R. ROGERS

2327. **Paper-disc electrophoresis in the determination of iso-electric points of amino acids.** N. C. Ganguli (Dept. of Appl. Chem., Univ. Coll. Sci. Technol., Calcutta, India). *Z. anal. Chem.*, 1957, **154** (3), 161–167 (in English).—The mobility and iso-electric points of various amino acids have been studied by paper-disc electrophoresis (*cf. Anal. Chem.*, 1956, **28**, 1499). The technique of the method is described. The mobility was studied at pH 2 to 7 by using citric acid - phosphate buffer, and at pH 8 to 10 by using KCl - NaOH buffer. With most amino acids, movement was from anode to cathode in a pH range of 2 to 5. Individual mobilities at different pH values are tabulated. In evaluating the iso-electric points, curves were plotted of mobility against pH. The relative mobilities, rate per hr. at pH 2 and pH 10, are tabulated, the distance moved by aspartic acid being taken as unity. W. J. WRIGHT

2328. **Specific determination of hydroxy-L-proline in biological materials.** A. E. Pasieka and J. F. Morgan (Lab. Hyg., Dept. Nat. Health Welfare, Ottawa). *Proc. Soc. Exp. Biol. Med.*, 1956, **92**, 96–99.—The sample is subjected to one-dimensional

descending paper chromatography, and the dried chromatograms are sprayed with ninhydrin. Rapid treatment with 1% $NaHCO_3$ and then with *N* HCl gives a stable pink derivative of hydroxyproline, which is not eluted with water, has an absorption max. at 550 to 560 mμ and fluoresces in u.v. light; 5 to 10 μg of the amino acid can be detected.
NUTR. ABSTR. REV.

2329. **Colorimetric determination of arginine with diacetyl monoxime.** R. Rendi (Ist. Fisiol. Gen., Rome). *Experientia*, 1957, **13** (1), 21–22 (in German).—Compounds containing a guanidine group give a positive reaction in the diacetyl monoxime test for urea (Wheatley, *Biochem. J.*, 1948, **43**, 420); thus arginine gives a colour with $\lambda_{max.} = 550$ mμ. The reaction has been applied to the quant. determination of arginine in proteins.
A. R. ROGERS

2330. **Development of analytical methods for some biological breakdown products of tryptophan.** D. Coppini (Inst. Pharm. Chem., Univ. Modena, Italy). *Mikrochim. Acta*, 1956, (12), 1818–1828 (in German).—Analytical methods for detecting small quantities of metabolic degradation products of tryptophan in blood and urine are reviewed. The detection and estimation of kynurenin, 3-hydroxykynurenin, anthranilic acid, 3-hydroxyanthranilic acid, kynurenic and xanthurenic acid are described. (42 references.) P. S. STROSS

2331. **A note on the determination of serum proteins by paper electrophoresis.** H. Roulet, J. A. Owen and C. P. Stewart (Dept. of Clin. Chem., Univ. of Edinburgh, Scotland). *Clin. Chim. Acta*, 1956, **1** (4), 417–420.—Three methods of evaluating scanner diagrams are compared. It is concluded that the method of choice is to cut out and weigh the portions corresponding to the different protein fractions. H. F. W. KIRKPATRICK

2332. **A quantitative method for the estimation of serum proteins by paper-disc electrophoresis.** N. C. Ganguli (Dept. of Applied Chem., Univ. Coll. of Science and Technol., Calcutta, India). *Clin. Chim. Acta*, 1956, **1** (4), 413–416.—A method is described in which 21-cm Whatman No. 1 filter-papers are used. The stained, separated protein fractions form arcs that are cut out, the dye is eluted and the colour measured photometrically.
H. F. W. KIRKPATRICK

2333. **Agar electrophoresis of serum proteins on Cellophane and polyester films.** K. V. Giri (Indian Inst. Sci., Bangalore, India). *J. Lab. Clin. Med.*, 1956, **48** (5), 775–778.—Electrophoresis of serum proteins is carried out in a thin layer of agar on a Cellophane or polyester film supported on a glass plate. After electrophoresis and staining, the dried agar and film may be cut into sections containing individual components and the concn. of dye measured directly or after elution. Good resolution of serum proteins is attained. The method is suitable for routine use and for radioactive-tracer analysis. W. H. C. SHAW

2334. **Procedure for the determination of proteins and similar compounds in large numbers of fractions.** Bengt v. Hofsten (Inst. Biochem., Univ. Uppsala, Sweden). *Acta Chem. Scand.*, 1956, **10** (9), 1432–1439 (in English).—In this relatively simple procedure, the ninhydrin reaction is used for the determination of nitrogenous matter present in small amounts in fractions obtained by chromatography, electrophoresis or counter-current extraction. The coeff. of variation based on eight determinations is $\simeq 1{\cdot}5\%$ for fractions containing $\simeq 3$ μg of N per ml. *Procedure*—Hydrolyse 0·1 ml of sample by heating at 120° for 10 to 24 hr. with constant-boiling HCl (0·5 ml) in a sealed ampoule. Open the ampoule, allow to evaporate overnight *in vacuo* over NaOH and dissolve the residue in H_2O (2 ml). Use a 1-ml aliquot to develop the colour with ninhydrin reagent according to the method of Moore and Stein (*cf. Anal. Abstr.*, 1955, **2**, 986) and measure the extinction at 570 mμ.
A. R. ROGERS

2335. **A study of the densitometric method of analysing [electrophoretic serum patterns on] filter-paper.** G. Klatskin, O. M. Reinmuth and W. Barnes (Yale Univ. Med. Sch., New Haven, Conn., U.S.A.). *J. Lab. Clin. Med.*, 1956, **48** (3), 476–490.—Sources of error in determining serum proteins by electrophoresis on filter-paper and subsequent staining with bromophenol blue or Amido black 10B are studied. Deviations from Beer's law obtained by direct scanning are minimised by using monochromatic light, although other factors, especially irregularities in dye-distribution, contribute to the errors. Application of factors to correct for the differences in dye-binding capacity of different proteins is unreliable. With a commercially available (in U.S.A.) compensating recording densitometer, stained serum patterns on filter-paper could be analysed directly and rapidly and with the minimum of scanning errors. W. H. C. SHAW

2336. **The estimation of dye in stained paper-electrophoresis strips [of proteins] using the "EEL" scanner.** R. L. Smith (Chase Farm Hosp., Enfield, Middx., England). *Clin. Chim. Acta*, 1956, **1** (2), 122–126.—The density readings obtained by scanning the stained paper-electrophoresis strips are not in linear proportion to the amount of stain in the protein. A method is described for calibration so that the readings can be plotted to correspond with the amount of dye actually present.
H. F. W. KIRKPATRICK

2337. **Applications of infra-red spectroscopy. The infra-red spectroscopy of steroids.** J. E. Page (Glaxo Lab. Ltd., Greenford, Middlesex, England). *Chem. & Ind.*, 1957, (3), 58–63.—This review deals with the recording of the i.r. spectra, the correlation of structure and stereochemical configuration with absorption, and the i.r. absorption of steroidal sapogenins. (46 references.) K. A. PROCTOR

2338. **Steroids. XXVI. Paper chromatography of neutral steroids.** J. Kučera, Z. Procházka and K. Vereš (Chem. Inst., Acad. Sci., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (1), 97–102.—The application of reversed-phase chromatography to neutral steroid compounds is described. Adsorption chromatography with light petroleum on unimpregnated paper and the use of some new detecting agents for steroid compounds are also described. J. ZÝKA

2339. **Spot test for 3-hydroxy-Δ^5-steroids.** R. F. Witter and S. Stone (Univ. Rochester, Rochester, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 156–157.—Steroids containing the 3-hydroxy-Δ^5 grouping give a characteristic pink colour when heated at

80° on paper that has been sprayed with phenol and then with $HClO_4$ and molybdic acid. The steroids can be detected at concn. as low as 5 μg per 10 μl. A keto group or a double bond in position 7 causes interference, and fructose and its derivatives give a purple colour. G. P. Cook

2340. **An improved method for the determination of total serum cholesterol.** D. M. Colman and A. F. McPhee (Radiation Lab., Univ. of California, Berkeley, U.S.A.). *Amer. J. Clin. Path.*, 1956, **26**, 181–186.—The lipids are extracted from the serum with ethanol - acetone. The extract is saponified with KOH for 30 min. at 37° and neutralised against phenolphthalein with HCl. Digitonin is added and the ppt., formed overnight, is washed and then treated with Liebermann - Burchard reagent. The soln. are kept at 25° for 32 min. and the colours measured at 645 mμ. R. S. Tonks

2341. **Evaluation of Silber - Porter procedure for determination of plasma hydrocortisone.** R. E. Peterson, A. Karrer and S. L. Guerra (Nat. Inst. of Health, Bethesda, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 144–149.—The following modifications were made to the Silber - Porter procedure (*J. Biol. Chem.*, 1954, **210**, 923)—(*a*) substitution of CH_2Cl_2 for $CHCl_3$ and its purification by silica gel, (*b*) the use of extraction procedures that did not require centrifuging, (*c*) the use of a smaller vol. of phenylhydrazine reagent which enables a smaller vol. of plasma to be used and also increases the sensitivity, (*d*) incubation of the phenylhydrazine reagent at room temp. For plasma hydrocortisone concn. of 5, 15 and 50 μg-%, precisions of ±5, ±1·5 and ±0·5%, respectively, were obtained.
G. P. Cook

2342. **Colour reaction between 17-ketosteroids and 3:5-dinitrobenzoic acid.** S. Cohen and A. Kaluszyner (Medical Corps, Israel Defence Forces, Israel). *Anal. Chem.*, 1957, **29** (1), 161–164.—In the presence of aq. alkali and under suitable conditions, 3:5-dinitrobenzoic acid gives a brilliant mauve colour with 17-ketosteroids. By carrying out the reaction in aq. methanol, both the blank value and interference from non-ketonic compounds can be kept low. The absorption max. of 550 mμ and extinction coefficients are practically identical for the four compounds tested. When applied to urine extracts, a mean recovery of 89·5% of added 17-ketosteroids was obtained. Reasonable agreement with other methods was reached.
G. P. Cook

2343. **Quantitative colorimetric estimation of oestradiol and oestrone in tissues.** F. Baum, E. Krekeler and J. Stengele (Univ. Frauenklinik, Würzburg, Germany). *Klin. Wochschr.*, 1957, **35** (2), 85–87.—Extract 20 to 50 g of tissue thoroughly with 75% ethanol. Heat the combined ethanol extracts with an equal vol. of 4% KOH soln. (aq.) under reflux in boiling water for 2 hr. At the same time heat the tissue residue with 50 ml of 0·5 *N* ethanolic KOH similarly. Combine the two soln., adjust to pH 6 with conc. HCl, filter and remove the ethanol *in vacuo*. Saturate the aq. hydrolysate with NaCl and shake out with 50, 30 and 20 ml of thiophen-free benzene. Wash the benzene soln. successively with 50 ml each of H_2O, 5% Na_2CO_3 soln. saturated with NaCl, and H_2O. Distil off the benzene, dissolve the residue in 100 ml of 70% methanol and shake with light petroleum (2 × 50 ml). Distil off the methanol, dissolve the residue in 100 ml of benzene and shake out the phenolic steroids with 50, 30 and 20 ml of 2 *N* NaOH. Acidify the combined NaOH extracts to pH 6 with conc. HCl, re-extract the steroids with 50, 30 and 20 ml of benzene and wash the benzene as before. Distil off the benzene, dissolve the residue in 4 ml of absolute methanol, add 0·165 g of Girard reagent T and reflux in boiling H_2O for 4 hr. Evaporate *in vacuo* to dryness, and dissolve the residue in 7 ml of buffer soln., pH 8·2, at 0° (1·95 g of 85% H_3PO_4, 0·992 g of boric acid, 0·96 g of glacial acetic acid, 160 ml of H_2O and 496 ml of 0·1 *N* NaOH). Extract with peroxide-free ether (2 × 10 ml), wash the ether with 2 ml of buffer soln., then with 2 ml of H_2O, dry the ether and distil to dryness. Dissolve the residue in 1 ml of absolute ethanol and to 0·7 to 0·9 ml in an ice bath add 2 ml of conc. H_2SO_4, heat in boiling H_2O for 6 min., then cool in the ice bath. Add 8 ml of 25% (v/v) H_2SO_4, heat in boiling H_2O for 6 min., and cool. Read the red colour at 512 mμ and obtain the oestrogen content (in μg) from a standard oestradiol curve. The recovery of added oestrogen was 70%. H. F. W. Kirkpatrick

2344. **Aldosterone. I. Isolation from adrenal cortical extract and a test for its identity.** A. G. Gornall and C. Gwilliam (Dept. of Path. Chem., Univ. of Toronto, Toronto, Canada). *Canad. J. Biochem. Physiol.*, 1957, **35** (1), 71–78.—Aldosterone was isolated by extraction with $CHCl_3$ and paper chromatography with three different solvent systems. The final material gave a positive blue tetrazolium and a negative Porter - Silber reaction, and in methanol soln. showed a symmetrical peak at 240 mμ. The hormone is estimated quant. by measuring the extinction in methanol at 235, 240 and 245 mμ of a soln. containing 1 to 13 μg of aldosterone per ml, and correcting the extinction at 240 mμ by subtracting half the sum of the other two extinctions. Measurement can also be made by means of the reaction with dinitrophenylhydrazine. After 5 min. at 20°, max. extinction is shown at 450 mμ, corresponding to the reaction of the Δ^4-3 ketone only; after 90 min. at 59°, the molar extinction coeff. is more than doubled and the peak shifts to 460 mμ. This reaction appears to be specific. H. F. W. Kirkpatrick

2345. **Chemical identification of aldosterone in post-operative urine.** J. G. Llaurado, R. Neher and A. Wettstein (Univ. of Otago, Dunedin, New Zealand). *Clin. Chim. Acta*, 1956, **1** (3), 236–241.—Urine (24-hr. specimens) was extracted with $CHCl_3$ at pH 1 and the $CHCl_3$ removed after washing with NaOH and water. Aldosterone was identified in a pooled extract, representing 80 specimens of post-operative (1 to 4 days) urine, after separation by chromatography, by conversion into the characteristic γ-lactone obtained in cryst. form.
H. F. W. Kirkpatrick

2346. **Determination of amylase activity in biological fluids.** H. V. Street and J. R. Close (Dept. of Pathol., Crumpsall Hosp., Manchester, England). *Clin. Chim. Acta*, 1956, **1** (3), 256–268.—In the method described, amylose is used as the substrate, and its prep. from starch, together with a test for purity, are given. The estimation uses 0·4 ml of serum and is completed in about 25 min.
H. F. W. Kirkpatrick

2347. **A rapid semi-quantitative method for the assay of tyrosinase activity.** J. A. Cooper and F. C. Brown (M. D. Anderson Hosp. and Tumour Inst., Houston, Texas, U.S.A.). *Clin. Chim. Acta*, 1956, **1** (4), 301–304.—A gel is prepared by boiling 2% Bacto-agar in 0·1 *M* phosphate buffer, pH 6·8. This can be stored indefinitely in the refrigerator. For use, the gel is reheated until solution is complete and an equal vol. of dopa soln. or tyrosine suspension (2 mg per ml) is added. A control soln., made with an equal vol. of water, is poured into Petri dishes to a depth of 5 mm and allowed to harden in the refrigerator. A razor blade is then passed diametrically across the dish and one half of the gel is removed, and replaced by pouring in substrate gel to the same depth and allowing to harden. Holes 4 mm in diam. are bored out and the test prep. is added with a micro-pipette. The plates are incubated at 38°. Tyrosinase activity is indicated in dopa plates by the formation of a pink coloration round the holes within 10 min., which subsequently becomes black (melanin). In tyrosine substrate the rings form much more slowly, but the reaction is more specific. Serial dilution makes the procedure semi-quant., the results with dopa being read after 30 min. H. F. W. KIRKPATRICK

2348. **Estimation of peptidase activity by quantitative paper chromatography.** R. J. Haschen (Physiol. Chem. Inst. der Martin Luther Univ., Halle-Salle, Germany). *Clin. Chim. Acta*, 1956, **1** (3), 242–255.—In the method described, the hydrolysis of leucylglycylglycine by serum is measured. The liberated amino acid is separated by paper chromatography, the ninhydrin - copper complex is formed, and eluted, and the colour is measured photometrically. Results from normal individuals and from patients with various diseases are tabulated. H. F. W. KIRKPATRICK

2349. **New assay method for tripeptidase.** M. London, A. Fine and P. B. Hudson (Inst. Cancer Res., Columbia Univ., New York, U.S.A.). *Arch. Biochem. Biophys.*, 1956, **64** (2), 412–422.—The colorimetric method described is based on the difference in colour produced between the copper complex of diglycylglycine (substrate) and that of Cu and the hydrolytic products. Enzyme (0·1 ml) and substrate (0·1 ml) soln. are incubated at 37° in a centrifuge tube for sufficient time to ensure about 50% hydrolysis of substrate. The reaction is then stopped with methanol (0·3 ml), and after further incubation for 5 min. the vol. is made up to 4 ml with sodium borate buffer. Freshly prepared $Cu_3(PO_4)_2$ gel (1 ml) is then added, and after 5 min. at room temp. excess of gel and denatured protein are removed by centrifuging. The extinction of the clear blue supernatant liquid is measured at 540 mμ in a 1-cm cell against water. A control is prepared by adding the enzyme after the methanol, and the percentage hydrolysis is determined from the difference in extinction between test and control soln. by means of a standard curve. The theory and kinetics of the reaction are discussed.
W. H. C. SHAW

2350. **Photometric determination of serum cholinesterase activity.** W. T. Caraway. *Amer. J. Clin. Path.*, 1956, **26** (8), 945–955.—The method is based on the decrease in absorbance of phenol red by the acetic acid liberated from acetylcholine by serum cholinesterase. Variations in initial absorbance, caused by selective binding of the indicator to serum proteins, were nullified by a calibration curve based on the ratio of final to initial absorbance. Pipette 5 ml of buffer - indicator mixture [anhydrous Na_2HPO_4 (6·65 g), KH_2PO_4 (0·43 g), and 10 ml of a 0·1% soln. of phenol red made up to 1 litre with water] into a cuvette. Add 0·5 ml of serum and 0·5 ml of a 15% soln. of acetylcholine bromide. Mix well and measure the absorbance (A_1) immediately at 540 mμ. Set aside for exactly 30 min. at 25° ± 1° and remeasure the absorbance (A_2). Calculate the ratio $A_2:A_1$ and read off units of cholinesterase activity from a calibration curve. Subtract the value determined for the control tube (in which 0·5 ml of water is substituted for the serum). R. S. TONKS

2351. **New method for determination of serum cholinesterase.** J. L. Luna Pérez (Clín. Quír., Fac. Med., Univ. Madrid). *Rev. Clín. Española*, 1956, **61**, 245–247.—Samples of blood (≃ 6 ml) were centrifuged at 2000 r.p.m. for 10 min. to obtain clear serum. To each of 11 test-tubes were added, in order, 1 ml of physiological saline soln., 1 drop of cresol red indicator, 1 ml of 0·01 *N* NaOH, 0·5 ml of acetylcholine chloride soln., 1 ml of prostigmine soln. and 0·25 ml of serum. One tube had no prostigmine, the other 10 increasing amounts from 0·5 to 5 μg. The tubes were incubated at 35° to 37° until the colour of the control soln. had changed from violet to canary yellow. Then in any tubes which were still intermediate in colour the reaction was considered to be inhibited. Greater or smaller amounts of prostigmine could be used if necessary, but in normal subjects tested the amounts of prostigmine capable of inhibiting serum cholinesterase always fell in the range 0·5 to 5 μg.
NUTR. ABSTR. REV.

2352. **Colorimetric measurement of serum glutamic oxalacetic transaminase.** P. Cabaud, R. Leeper and F. Wróblewski. *Amer. J. Clin. Path.*, 1956, **26** (9), 1101–1105.—Serum glutamic oxalacetic transaminase mediates the transfer of the α-amino N of aspartic acid to α-oxoglutaric acid, resulting in the synthesis of glutamic and oxalacetic acid. The oxalacetate formed is converted into pyruvate, which is estimated colorimetrically. Add serum (0·5 ml) and water (0·5 ml) to each of two test-tubes. At zero time add 0·5 ml of enzyme substrate to each tube [DL-aspartic acid (2·66 g), KH_2PO_4 (2·0 g) and α-oxoglutaric acid (0·6 g) dissolved in 100 ml of water, with the pH adjusted to 7·4 with KOH]. Add immediately one drop of trichloroacetic acid soln. (100%, w/v) (**I**) and one drop of aniline citrate (5 ml of aniline added to 5 g of citric acid in 5 ml of water) (**II**) to one of the tubes, to serve as a blank. Incubate the tubes for 20 min. at 26° and add one drop of each of **I** and **II** to the second tube, shake the tubes and allow them to stand for 20 min. Add 0·5 ml of dinitrophenylhydrazine reagent (100 mg of 2:4-dinitrophenylhydrazine in 20 ml of conc. HCl and 80 ml of water) to both tubes, mix the contents well and allow to stand for 5 min. Add 2 ml of toluene to each tube, shake vigorously and centrifuge for 5 min. Remove 1 ml of toluene from the top layer of each tube and place in separate colorimetric tubes. Add 3 ml of KOH soln. (2·5 g of KOH in 100 ml of 95% ethanol) to each tube and shake. Measure the colour formed at 490 mμ with the blank set at 100% transmittance.
R. S. TONKS

See also Abstracts 2208, 2234, 2235, 2423, 2462, 2463, 2470.

Drugs

2353. **A new reaction for the group detection of alkaloids.** G. Ya. Gornȳĭ. *Ukr. Khim. Zhur.*, 1955, **21** (6), 761–762; *Ref. Zhur., Khim.*, 1956, Abstr. No. 47,364.—For the spot detection of alkaloids, H_2PtI_6 is used, made by adding a 10% soln. of H_2PtCl_6 (0·1 ml) to a 2% soln. of KI (1 ml). To a filter-paper soaked in H_2PtI_6, transfer one drop of the washed chloroform, ether or ethanolic alkaloidal extract; in the presence of alkaloids a clear violet, blue - violet or red - brown spot appears, which is not removed after 1 or 2 min. washing with water (with the exception of caffeine). Appended is the sensitivity of the reaction for 24 of the most important alkaloids. C. D. KOPKIN

2354. **Polarographic determination of morphine in poppy capsules.** J. Holubek (Inst. Heilpflanzenforsch., Prague, Czechoslovakia). *Pharm. Zentralh.*, 1956, **95** (11), 435–436.—The extraction procedures of Pfeifer and Keller (*Anal. Abstr.*, 1957, **4**, 645) and Holubek (*Anal. Abstr.*, 1956, **3**, 809) are compared. In contradiction to the findings of the former authors, results differing by only 5 to 10% were obtained with both methods of extraction and the polarographic waves were substantially similar. The acid alumina method is quicker and requires less of the sample. P. S. STROSS

2355. **Analysis of narcotics. IV. Non-aqueous titration.** Hiroshi Sakurai and Etsuo Awada (Shinagawa Plant, Sankyo Co. Ltd., Shinagawa-ku, Tokyo). *J. Pharm. Soc. Japan*, 1956, **76** (9), 1026–1030.—The potentiometric titration of codeine and thebaine with toluene-*p*-sulphonic acid (**I**) in $CHCl_3$, dioxan, a mixture of ethanediol and *iso*propyl alcohol (1 + 1) (**II**) and glacial acetic acid was investigated. The titration curve becomes less distinctive with these solvents in the order given. The effect of expansion of various solvents was also examined; it is minimised by the use of **II**. Some alkaloids, including morphine, codeine, thebaine, dihydrocodeine, papaverine, dihydrohydroxycodeine and hydrocotarnine (≃ 20 mg) are dissolved in $CHCl_3$ (20 ml) and phenol (2 ml) and titrated with 0·005 *N* **I** in **II**, with methyl orange as indicator. K. SAITO

2356. **Determination of micro quantities of alkaloids by the amperometric method. V. Titration of codeine and Dionin [ethylmorphine hydrochloride] in hydrochloric acid solution with tungstosilicic acid.** Tadahiko Ogawa (Electrochem. Lab., Fac. of Engng, Yokohama Univ., Minami-ku, Yokohama). *J. Electrochem. Soc. Japan*, 1956, **24** (10), 476–479.—The amperometric titration of codeine phosphate (4 to 42 mg) (**I**) and ethylmorphine hydrochloride (**II**) (8 to 40 mg) was studied with tungstosilicic acid (**III**) in dil. HCl soln. Neither **I** nor **II** shows a reduction wave at $> -0{\cdot}90$ V *vs.* the S.C.E. The concn. of HCl must be kept between 1·0 and 4·0 *N* throughout the titration. The sample is dissolved in HCl (4·0 *N*, 5 to 10 ml) and titrated with aq. **III** soln. (28 mg per ml) at − 0·70 V. Interference results from the presence of those substances that react with **I**, **II**, **III** or HCl, or show a reduction wave under the given conditions. The error is ≃2%.

VI. Titration of papaverine, ethylmorphine hydrochloride and veratrine in a neutral solution with tungstosilicic acid. Tadahiko Ogawa. *Ibid.*, 1956, **24** (11), 521–524.—Similar experiments were carried out with papaverine hydrochloride (**IV**), **II** and veratrine sulphate (**V**) in a neutral soln. containing KNO_3. The reduction waves of **IV** (< 40 mg) and **V** (< 40 mg) start at − 0·80 and − 1·20 V, respectively. The wave height at −0·75 V is proportional to the concn. of **III** in the presence of $> 0{\cdot}05$ *M* KNO_3. The sample is dissolved in 0·42 *M* KNO_3 (10 ml) and titrated with aq. **III** soln. (28 mg per ml) at − 0·75 V. The error is ≃2%. Salts having their reduction wave at < − 0·80 V can also be used for the supporting electrolyte. K. SAITO

2357. **The microscopic examination and identification of ergot alkaloids. II.** J. Kolšek (Inst. for Organ. Chem., Univ. Ljubljana, Yugoslavia). *Mikrochim. Acta*, 1956, (12), 1783–1787 (in German).—The alkaloids ergosinine, ergocorninine and ergocryptinine are studied by Kofler thermomicro methods. The crystals obtained by crystallisation in several solvents are described, and crystallographic data and melting-points of the alkaloids and some derivatives are given. The identification of the alkaloids from these data is possible. P. S. STROSS

2358. **Spectrophotometric determination of cocaine, procaine and tetracaine [amethocaine].** M. J. Pro, R. A. Nelson, W. P. Butler and A. P. Mathers (Alcohol and Tobacco Tax Div. Lab., Internal Revenue Service, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1956, **39** (4), 957–964.—Absorption curves for cocaine, procaine and tetracaine in NaOH between 200 and 340 mμ are presented. The sample (100 mg) is diluted with 0·03 *N* NaOH to 200 ml. Of this, a 5-ml aliquot is heated at 100° (15 min.) with 10 ml of 0·3 *N* NaOH, cooled, and diluted to 100 ml with H_2O. The extinction is read at 226 mμ for cocaine, 264 mμ for procaine, and 280 mμ for tetracaine against the 0·03 *N* NaOH solution. Then cocaine (p.p.m.) = $42{\cdot}45\,A_{226} - 0{\cdot}74\,A_{264} - 12{\cdot}00\,A_{280}$; procaine (p.p.m.) = $-\,2{\cdot}49\,A_{226} + 37{\cdot}59\,A_{264} - 24{\cdot}42\,A_{280}$; and tetracaine (p.p.m.) = $1{\cdot}06\,A_{226} - 27{\cdot}01\,A_{264} + 36{\cdot}02\,A_{280}$, where A is the extinction at the wavelength named. The infra-red spectra (illustrated) serve for identification. A. A. ELDRIDGE

2359. **Determination of the digitoxin content of *Digitalis purpurea*.** E. H. B. Sellwood (Allen & Hanburys Ltd., Ware, Herts., England). *J. Pharm. Pharmacol.*, 1956, **8** (12), 1061–1071.—A new paper-chromatographic and colorimetric method of estimating the digitoxin content of digitalis leaf is described. The powdered leaf (20 g) mixed with 20 ml of water is allowed to undergo autolytic fermentation at 37° for 68 hr., then extracted with ethanol (70%); the ethanol is evaporated and the aq. residue is extracted with 1:2-dichloropropane. The extract is washed with aq. Na_2CO_3 soln. and evaporated to dryness *in vacuo*, and the residue, dissolved in *iso*propyl alcohol, is chromatographed (descending technique) on Whatman No. 1 paper, with CCl_4 - ethanol - H_2O (1:1:1). The digitoxin zones, detected by spraying a corresponding chromatogram with trichloroacetic acid in $CHCl_3$, were cut out, the digitoxin was allowed to react with a solution of xanthhydrol in glacial acetic acid containing a trace of HCl, and the colour was compared with a standard. The xanthhydrol reagent is 4·5 times as sensitive as the Na picrate reagent. The chromatography separates gitoxin, gitoxigenin, digitoxin and digitoxigenin, and recoveries of digitoxin are 91 to 95%; digitoxose does not interfere.

Optimum conditions for the fermentation are described and it is shown that digitoxin is released from its complexes, but not broken down.
P. S. Stross

2360. **The reaction between digitoxin and 3:5-dinitrobenzoic acid.** D. H. E. Tattje (Pharmacog. Lab., Univ. Groningen, Holland). *J. Pharm. Pharmacol.*, 1957, **9** (1), 29–34.—The optimum concentrations and conditions for carrying out the Kedde reaction of digitoxin (**I**) with 3:5-dinitrobenzoic acid (**II**) have been studied. The reaction should be performed at a temp. of $<20°$. Ethanol is preferred to methanol or *n*-propanol as solvent. A diminution in the ethanol content of the reaction mixture exerts only slight influence on the max. extinction and the rate of reaction. Soln. of **II** in ethanol may be stored for several days without deterioration, but the (alkaline) blank soln. must be freshly prepared for each estimation. With the recommended procedure, the Beer - Lambert law is obeyed and $E^{1\%}_{1\,cm}$ for **I** is $111 \pm 1{\cdot}5$. *Procedure*—Dissolve the sample ($<0{\cdot}8$ mg of **I**) in ethanol (4 ml), mix with a 2% soln. of **II** in ethanol (5 ml), add 1·5 *N* NaOH (1 ml) and after exactly 4 min. compare the extinction with that of a simultaneously prepared reagent blank in 1-cm cells at 540 mμ.
A. R. Rogers

2361. **Ultra-violet spectrophotometric determination of tetracycline in cultures.** Shigeharu Inoue, Teiichiro Itô and Hiroshi Ogawa (Res. Lab., Meiji Seika Co. Ltd., Kawasaki). *J. Agric. Chem. Soc. Japan*, 1956, **30** (10), 591–596.—The culture for the preparation of tetracycline (**I**) contains an unknown substance which absorbs u.v. light. An empirical formula for the u.v. spectrophotometric determination of **I**, both in the presence and absence of chlortetracycline (**II**), is proposed. The concn. of **I** chloride (5 to 25 μg per ml) in the absence of **II** is expressed as C (mg per ml) $= 0{\cdot}1159k_{355} - 0{\cdot}0869k_{340} - 0{\cdot}0290k_{400}$, where k_{355}, etc., is the extinction of the sample soln. in 0·1 *N* H_2SO_4 at 355 mμ, etc. (thickness of the layer, 1 cm). In the presence of **II**, it is expressed as $C = 0{\cdot}185k_{350} - 0{\cdot}101k_{330} - 0{\cdot}0673k_{310} - 0{\cdot}0168k_{390}$, whereas that of **II** as C (mg per ml) $= 0{\cdot}1485k_{330} + 0{\cdot}295k_{370} - 0{\cdot}370k_{350} - 0{\cdot}0733k_{390}$. The results agree well with those by bio-assay.
K. Saito

2362. **Chloramphenicol benzoate. III. Methods of estimation of relative potency of chloramphenicol and its esters.** Motohiko Maruyama and Yoshio Suzuki (Takamine Lab., Sankyo Co. Ltd., Shinagawa-ku, Tokyo). *Ann. Rep. Takamine Lab.*, 1955, **7**, 150–158.—The relative efficacies of chloramphenicol (**I**), its benzoate (**II**) and palmitate (**III**) were examined therapeutically, according to Akiba and Nagabayashi's formula (curative ratio $= 100b/a + 100(m-n)(a-b)/a(p-n)$, where a is the no. of animal members of the treated group; b is the no. of survivors; m is the average time (days) of survival; n is the average time (days) of survival for the control group; and p is the no. of days of observation). Mice of German breed were injected with various amounts of **I**, **II** or **III** for defence against infection with *Salmonella enteriditis*. When the curative ratio was plotted against the log. of the dose, a linear relationship was obtained. The curative ratio as a criterion for efficacy is discussed. **III** appears to be slightly inferior to **I** and **II**. The application of Bülbling and Burn's equation (*Quart. J. Pharm. Pharmacol.*, 1938, **11**, 67) was also studied and found to be as useful as Akiba's.
K. Saito

2363. **Volatile organic acids of tobacco smoke.** D. A. Buyske, P. Wilder, jun., and M. E. Hobbs (Duke Univ., Durham, N.C., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 105–108.—The qualitative identification and quant. determination of the steam-volatile monocarboxylic acids, present in the smoke from three major types of tobacco, are reported. The methods used involved partition chromatography on buffered silicic acid columns, paper chromatography and i.r. and u.v. spectrometry. Acetic acid accounted for 50% and formic acid for 25% of the total acidity found, and *iso*butyric, *iso*valeric, benzoic and an unknown acid were also determined. The concn. of the homologous series from propionic to *n*-decoic acid varied with the type of tobacco. About 90% of the acids were accounted for and the remaining 10% included at least five additional acids.
G. P. Cook

2364. **Scheme for the identification of some barbiturates.** M. A. Robles (Lab. Artsenijbereidkunde, Univ., Amsterdam). *Pharm. Weekbl.*, 1957, **92** (2), 41–46.—The barbiturates are identified in groups depending on the colours given in the following tests. (*a*) Heat the sample (50 mg) with vanillin (50 mg) and conc. H_2SO_4 (15 drops) for 5 or 10 min. on a bath of boiling water. (*b*) Add ethanol (2 ml) to (*a*). (*c*) Add an equal vol. of water to (*b*) and allow to stand for 15 min. In addition, tests for an N-methyl group and for a phenyl group, and microscopic examination of the crystals obtained with the reagent of Lüdy-Tenger (*Pharm. Acta Helv.*, 1944, **19**, 385) are recommended. (13 photomicrographs.)
A. R. Rogers

2365. **Determination of barbiturates. I. Alkalimetry, argentimetry and ultra-violet spectroscopy.** Takanobu Itai and Takuma Oka. *Bull. Nat. Hyg. Lab., Tokyo*, 1955, (73), 131–135.—The three assay methods are compared. For alkalimetry an indicator consisting of a mixture of phenolphthalein and Nile blue is better than thymolphthalein.

II. Titration in non-aqueous solution. Takanobu Itai and Takuma Oka. *Ibid.*, 1955, (73), 137–139.—Pentobarbitone, allobarbitone and amylobarbitone can be separately titrated in a benzene - methanol mixture with Na methoxide.
N. E.

2366. **The determination of isoniazid in pharmaceutical preparations containing sodium *p*-aminosalicylate.** B. W. Mitchell, E. A. Haugas and C. S. McRoe (Anal. Div., Smith and Nephew Research Ltd., Hunsdon, England). *J. Pharm. Pharmacol.*, 1957, **9** (1), 42–45.—In this rapid and accurate method for the determination of isoniazid (**I**) in the presence of 4-aminosalicylic acid and its derivatives, the hydrazine group of **I** is quant. reduced, by a zinc - copper couple in KOH soln., to NH_3, which is determined by titration. *Procedure*—Shake powdered Zn (10 g), H_2O (25 ml) and $CuSO_4$ soln. (25%) (10 ml) in a 1-litre flask until the supernatant liquid is nearly colourless. Wash by decantation with H_2O (3 × 50 ml). Add the sample (containing 50 to 150 mg of **I**) together with KOH soln. (2·5%) (400 ml) and distil into 0·1 *N* H_2SO_4 (25 ml), until 100 or 150 ml has been collected (15 to 30 min.). Back-titrate the excess of acid with 0·05 *N* NaOH, with methyl red screened with methylene blue as indicator.
A. R. Rogers

2367. **The photometric determination of potassium 8-hydroxyquinoline sulphate in ointments and suppositories.** H. Klaus (Inst. f. Veterin.-Pharmakol. und Toxicol., Karl-Marx Univ., Leipzig, Germany). *Pharm. Zentralh.*, 1956, **95** (11), 429–432.—The method is based on the formation of the blue-green compound between potassium 8-hydroxyquinoline sulphate (**I**) and a ferric salt. The calibration curve is prepared with solutions containing between 10 and 25 mg of **I** per 100 ml of water, to which is added a measured amount of aq. $Fe(NO_3)_3.9H_2O$ soln. Since **I** is water-soluble and insoluble in ether, it is extracted from ointments and suppositories by dissolving them in ether and extracting the ethereal solution with successive portions of water. The standard deviation in the analysis of ointments is $\pm 4\%$ and in the analysis of suppositories $\pm 2{\cdot}5\%$.
P. S. Stross

2368. **Chromatography in the separation of antiseptic quaternary ammonium compounds.** I. Garcia and J. Couerbe (Fac. Méd. et Pharmacie, Algiers). *Chim. Anal.*, 1956, **38** (12), 432–434.—In an investigation of long-chain quaternary ammonium compounds, used as antiseptics, an ascending chromatographic process was examined. The compounds were: benzyldodecyldimethylammonium chloride, benzyldimethyl*iso*octylphenoxyethoxyethylammonium chloride, benzalkonium chloride, domiphen bromide, benzyldodecyldimethylammonium bromide, cetrimide and triheptylmethylammonium methylsulphate, all within concn. limits of 5 to 15 μg. The most suitable solvent is pyridine-benzene-acetic acid-H_2O (80:80:2:8). Chromatography was carried out at 22°. Temp. had a great influence on R_F. The chromatograms were taken after 14 to 15 hr. when the solvent had traversed 45 to 47 cm. To detect the spots, the chromatogram was sprayed with Na_2CO_3 solution (5%). After drying, it was sprayed with a solution of bromophenol blue (0·02%), which coloured the spots blue on violet. They were intensified by washing with running H_2O to remove excess of bromophenol blue and impurities and finally dried in warm air. The spots may also be revealed by Nessler reagent (white on red), or $K_3Fe(CN)_6$ and $FeSO_4$ (yellow on blue). W. J. Wright

2369. **Limit tests for impurities [in drugs]. IV. Investigations into the reproducibility of precipitation. 3.** J. Zimmermann, E. Krogh-Svendsen and F. Reimers (Dalgas Boul. Pharm., Copenhagen, Denmark). *Anal. Chim. Acta*, 1957, **16** (1), 6–21 (in English).—In the pptn. of trace quantities of SO_4^{2-} the reproducibility of the turbidities obtained is influenced by the concn. of the $BaCl_2$ soln., the use of seeding with $BaSO_4$ suspensions, the addition of ethanol and other factors. The pptn. of Ba^{2+} with H_2SO_4 and of Ca^{2+} with ammonium oxalate are affected similarly. These reactions have been investigated in the presence of various salts and acids, and standard conditions for limit tests are proposed. W. C. Johnson

2370. **Some statistical aspects of the analytical control and standardisation of tablets.** A. R. Rogers (School of Pharm., Brighton Tech. College, Sussex, England). *J. Pharm. Pharmacol.*, 1956, **8** (12), 1103–1113.—Statistical methods are applied to the variation in weight of tablets during manufacture. Methods of control, based on the standard deviation and weight range, to be applied by the manufacturer and by the independent analyst are suggested and compared with accepted methods and sequential analysis tests. The statistical aspect of the B.P. disintegration test is discussed. P. S. Stross

2371. **Recent applications of the Reinsch test.** M. Lessa Bastos (Inst. Médico Legal, Rio de Janeiro, Brazil). *Arquiv. Bromatol.*, 1956, **4** (1), 5–15.—Applications of the Reinsch test in toxicological and food chemistry, and the identification of trace elements deposited on the copper foil by chemical and by spectrographic methods are reviewed.
L. A. O'Neill

See also Abstracts 2296, 2297, 2299, 2402, 2403, 2404, 2405.

Food

2372. **Determination of composition of food and fodders. IV. Determination of reducing sugars by Lane-Eynon's method with a magic-eye titrator.** Makoto Kandatsu and Tamio Satow (Dept. Agric. Chem., Fac. Agric., Tokyo Univ., Hongo, Tokyo). *J. Agric. Chem. Soc. Japan*, 1956, **30** (6), 322–326.—The potentiometric titration of reducing sugars with Fehling soln. was studied by the use of a magic-eye in place of a potentiometer. In order to increase the linearity of the indication, a current feed-back circuit was employed by the use of a resistance, between the cathode of the amplifier and the plate of the rectifier, as "cathode follower." Fehling soln. *A* and *B* (5 ml of each) are added to a vessel containing copper electrodes, and the sample soln. is added gradually to the vessel, kept at boiling temp. The magic-eye closes with addition of the sample and shuts at the end-point. The results agree well with those obtained by the use of methylene blue as internal indicator. K. Saito

2373. **Japanese persimmon. II. Determination of total sugar in materials containing fructose.** Kiyoshi Aso and Toshiyuki Watanabe (Dept. Agric. Chem., Fac. of Agric., Tohoku Univ., Kitaroku-bancho, Sendai). *J. Agric. Chem. Soc. Japan*, 1956, **30** (4), 191–195.—The hydrolysis of fructose (**I**) was studied under various conditions of acid concn. and time of reaction. **I** does not undergo hydrolysis when heated in 0·1% HCl for 30 min. (the conditions for the hydrolysis of sucrose), but is converted into hydroxymethylfurfuraldehyde (**II**) when heated in 2·5% HCl for 2·5 hr. Since **II** is less reducing than **I**, low values result in the determination of total sugar by the Willstätter-Schudel method. Glucose, xylose and arabinose remain unchanged under the given conditions. The extent to which **I** is converted into **II** depends on the concn. of HCl and time of heating; but this effect was not completely eliminated whatever the conditions of hydrolysis. K. Saito

2374. **Quantitative chromatographic procedure for determining dextrose in sugar mixtures.** E. J. McDonald (Nat. Bureau of Standards, Washington, D.C., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 32–34.—Dextrose is separated from other sugars by paper chromatography and is then transferred from the chromatogram to glass-fibre paper by elution with water. The dextrose can be determined in the presence of the fibre by the method of Somogyi (*J. Biol. Chem.*, 1945, **160**, 61), with slight modifications. Good reproducibility is attained when the method is applied to the determination of dextrose in honey. G. P. Cook

2375. **Quantitative determination of reducing sugars and sucrose separated by paper chromatography.** R. S. Shallenberger and R. G. Moores (General Foods Corp., Hoboken, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 27–29.—The sugars are separated and detected by normal chromatographic procedure and are determined by treating the cut-out spots with Nelson's reagent (*J. Biol. Chem.*, 1944, **153**, 375) and measuring the absorbance at 500 mμ. A suitable correction is applied to allow for the interference caused by the paper. Sucrose is determined in the same way after hydrolysis with invertase. The standard deviation is < 5 μg in the range from 10 to 200 μg of sugar. The method is applicable to the analysis of plants and foods. G. P. Cook

2376. **Selective adsorption method for determination of the sugars of honey—correction.** J. W. White, jun. (Eastern Utilisation Res. Branch, Agric. Res. Service, Philadelphia, Pa., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1956, **39** (4), 1016.—A corrected version of the data for the Shaffer-Somogyi titration of hydrolysed sucrose solutions (*cf.* White and Maher, *Ibid.*, 1954, **37**, 466) is given, and the use of these results in determining the sucrose present in the eluate is described. A. A. Eldridge

2377. **Determination of dry matter and starch content of potatoes.** A. Metzger (Eidg. Agrik.-chem. Versuchsanstalt, Liebefeld, Bern). *Mitt. Lebensmitt. Hyg., Bern*, 1956, **47** (5), 344–350.—The distribution of dry matter and starch content in fresh and dried potatoes is observed and methods are described for the determination of these two components. Potatoes (2 to 4 kg) are chopped finely and 500 g of the resulting mix is analysed. A 20-g portion is hydrolysed with 1·315% HCl for 15 min. on a water bath and the sugars produced are determined by polarimetry (error 0·58%). The dry matter is determined by heating another 20-g portion of the mix for 6 hr. at 60° and 5 hr. at 105° (error 0·69%). A mathematical expression is derived from graphs for the calculation of percentage starch and dry matter. D. P. Felix

2378. **Protein precipitants for the determination of true protein in food and fodders. Precipitation of proteins with trichloroacetic acid in ten per cent. sodium chloride solution.** Makoto Kandatsu, Tadahiko Yasui and Noriko Yamamoto (Dept. Agric. Chem., Fac. Agric., Tokyo Univ., Hongo, Tokyo). *J. Agric. Chem. Soc. Japan*, 1956, **30** (6), 316–321.—The pptn. of glycinin and edestin with trichloroacetic acid (**I**) was studied in 10% NaCl soln.; it is quantitative, provided that the concn. of **I** in the precipitating soln. is kept $>2·5\%$, and that in the washing soln. $>1·0\%$. K. Saito

2379. **The putrefactive products of fish tissue. I. The turbidity in Dyer's colorimetric determination of trimethylamine.** Kaname Saito and Muneo Sameshima (Fac. Fisheries, Kagoshima Univ., Kamiarata-cho, Kagoshima). *J. Agric. Chem. Soc. Japan*, 1956, **30** (8), 531–535.—In the colorimetric determination of trimethylamine in fish tissue with picric acid (Dyer, *J. Fish. Res. Bd. Can.*, 1945, **6**, 351), a marked turbidity appears, which increases with time. This turbidity is dependent on the pH value of the sample (it does not appear with samples of pH <5 or >8) and was found to be due to the presence of cadaverine. It can be removed by the addition of Hg^{2+}. The sample soln. is made neutral with 10% Na_2CO_3 soln., then treated with a few drops of 25% mercuric acetate soln. and filtered. The colorimetric determination is made on the filtrate. K. Saito

2380. **Analysis of fresh meat.** E. J. Benne, N. H. Van Hall and A. M. Pearson (Mich. Agric. Exp. Sta., East Lansing, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1956, **39** (4), 937–945.—The prepared meat is rapidly passed through a grinding mill equipped with a fine-mesh cutting plate and a fixed divider which rejects half the ground sample, the remainder being reground and divided until a homogeneous sample results. For the determination of ash, the sample, dried at 100° to 105°, is ignited at a temperature gradually raised to 550°. The crude fat is determined by extracting the dried sample, contained in a Gooch crucible with asbestos mat, in a Bailey-Walker extraction flask. Moisture is determined by heating at 100° to 105° for 5 hr. (fat meat) or for 24 hr. (lean meat). For Kjeldahl digestion the sample is conveniently weighed on a tared piece of parchment paper, the meat being then rolled in the paper and the whole digested. The procedures are described in detail. A. A. Eldridge

2381. **Paper-chromatographic detection of sucrose in milk.** A. Castiglioni and R. Pilleri (Inst. f. Warenkunde, Univ. Turin, Italy). *Z. anal. Chem.*, 1957, **154** (3), 187–188.—Paper chromatography has been employed to separate lactose and sucrose. Whey (0·1 to 0·3 ml) prepared by acidifying milk with acetic acid, is treated on a strip of Whatman paper No. 1 with butanol-ethanol (95%)-H_2O (50:10:40). After 16 to 20 hr., the strip is withdrawn, dried, and sprayed with a soln. of 0·5 g of 1-naphthylamine in 50 ml of ethanol (95%) and 2·5 ml of conc. HCl. If sucrose is present, it appears as a brown spot after drying, easily distinguishable from the rose-coloured spot given by lactose. W. J. Wright

2382. **Procedures for making acidity determinations of fluid dairy products.** F. J. Doan, B. L. Larson, W. C. Winder and J. G. Leeder (American Dairy Sci. Ass., 32, Ridgeway Circle, White Plains, N.Y., U.S.A.). *J. Dairy Sci.*, 1956, **39** (10), 1384–1385.—This report of the Subcommittee for Recommended Uniform Procedures, A.D.S.A., gives details of a modified spot-plate method and a centrifuge method for determining the acidity of milk. W. H. C. Shaw

2383. **The influence of several factors on the protein and solids-not-fat content in the milk of Jersey cows.** L. W. Specht, J. R. Brunner, D. E. Madden and N. P. Ralston (Michigan State Univ., East Lansing, U.S.A.). *J. Dairy Sci.*, 1956, **39** (10), 1337–1341.—The effects of milking-time, herd differences, season and stage of lactation on the protein and solids-not-fat contents were studied on two herds over a 15-month period. Equations are given for estimating the solids-not-fat content from the percentage of fat or (more reliably) from the protein content. W. H. C. Shaw

2384. **Penicillin in milk. I. The rapid routine assay of low concentrations of penicillin in milk.** N. J. Berridge (Nat. Inst. Res. Dairying, Reading Univ., England). *J. Dairy Res.*, 1956, **23** (3), 336–341.—A collaborative study is made of the

rapid simple method described. Penicillin is determined by mixing the milk sample with an actively growing culture of *Streptococcus thermophilus* (N.I.R.D. no. 7), selected for rapid growth and high sensitivity to penicillin. Bromocresol purple (0·05%) is added, and the acidity produced after incubation for 2·5 hr. at 45° is estimated from the colour in comparison with standards containing concn. of penicillin corresponding to 0·01 to 0·08 i.u. per ml in the sample. Controls treated with penicillinase ensure the specificity of the test. A shortened incubation time (30 min.) gives a more rapid but slightly less sensitive test. The significance of the penicillin levels found is discussed.

II. The incidence of penicillin. N. J. Berridge. *Ibid.*, 1956, **23** (3), 342–347. Results of tests on 5000 samples of milk from eight areas in England and Wales are summarised. A significant proportion of individual samples contained > 0·04 i.u. per ml, but bulk milk rarely contained >0·1 i.u. per ml. The effect of such levels on cheese-making is discussed. W. H. C. SHAW

2385. **Detection and determination of diacetyl in butter produced in the province of Perugia.** M. Brogioni (Lab. Prov. d'Igiene e Profilassi, Perugia, Italy). *Ann. Falsif.*, 1955, **48**, 56–62.—The method of Pien (*Ibid.*, 1948, **41**, 529) in which diphenylquinoxaline is used was found to be the best for small quantities of diacetyl in butter. W. J. WRIGHT

2386. **Detection and determination of diacetyl in Italian butter.** M. Brogioni (Inst. Chim. Bromatol., Univ. Perugia, Italy). *Ann. Falsif.*, 1956, **49**, 499–501.—The method used previously (*Anal. Abstr.*, 1957, **4**, 2385) has been applied to other Italian butters. W. J. WRIGHT

2387. **Vacuum salt for butter and cheese making.** British Standards Institution (2 Park St., London). B.S. 998: 1957, 14 pp.—Revised specifications are given for chemical purity, and limits of Fe and SO_4^{2-} in both dried and undried vacuum salt, and a new clause on packaging and storing is included. The description, sampling, moisture content, limits of impurities and fineness of grain of dried salt are prescribed, and methods are given for determining these properties, also for chemical purity, alkalinity, matter insoluble in water, SO_4^{2-}, As, Ca and Mg; limit tests for Fe, Cu and Pb, and rapid sorting tests for SO_4^{2-}, Ca and Mg are described. O. M. WHITTON

2388. **Lactose crystallisation in ice-cream. II. Factors affecting rate and quantity.** T. A. Nickerson (California Univ., Davis, U.S.A.). *J. Dairy Sci.*, 1956, **39** (10), 1342–1350.—The polarimetric method of Sharp and Doob (*J. Dairy Sci.*, 1941, **24**, 589) for determining the α- and β-lactose contents of dry milk products is modified for use with ice-cream containing a standardised amount (15%) of sucrose. The rate of crystallisation of lactose from ice-cream under different conditions is studied. W. H. C. SHAW

2389. **The microchemical analysis of wine. The determination of the specific gravity, alcohol and solids.** G. Gorbach and H. Pfrimer (Inst. Biochem. Tech. and Nutrition, Tech. High School, Graz, Austria). *Mikrochim. Acta*, 1956, (12), 1774–1782 (in German).—Methods that require only 15 to 150 ml of wine and one-quarter to one-sixth of the time necessary for the official methods, and of an equal or greater accuracy, are described. The sp.gr., alcohol content and solids can be determined in succession on the same sample by using Pregl weight-pipettes and the Gorbach micro-distillation apparatus. The solids content may also be calculated by the use of tables from the sp.gr. of the soln. remaining after distillation. If the sp.gr. only is required, this may be determined on 2 to 5 ml of wine by a hydrostatic method. P. S. STROSS

2390. **The mechanism of the production of the aroma of black tea. II. Determination of volatile carbonyl compounds in black tea.** Toshio Nakabayashi (Fac. Agric., Shizuoka Univ., Iwata, Shizuoka Prefecture). *J. Agric. Chem. Soc. Japan*, 1956, **30** (10), 641–644.—The determination of total carbonyl compounds (cinnamaldehyde, perillaldehyde, citral, benzaldehyde, etc.) in black tea was studied by distillation, followed by iodimetric titration in the presence of $NaHSO_3$. No significant amount of carbonyl compound is produced from catechin at < 102°. The sample (5 g) is mixed with 150 ml of water and distilled over an oil bath at 150°. The distillate is collected in two flasks in series containing 0·75 *M* $NaHSO_3$ (10 and 5 ml) and titrated with 0·01 *N* iodine soln. K. SAITO

2391. **Free amino acid in tea leaf. II. Determination of asparagine and aspartic acid.** Zenji Nagashima and Muneyuki Nakagawa (Fac. Agric., Shizuoka Univ., Iwata, Shizuoka Prefecture). *J. Agric. Chem. Soc. Japan*, 1956, **30** (7), 410–413.—The determination of asparagine (**I**) and aspartic acid (**II**) was studied by bio-assay with *Leuconostoc mesenteroides*. **I** and **II** are separated with Amberlite XE-114 (Cl); **I** (in the eluate) is converted into **II** by heating with 4 *N* HCl in a sealed tube at 120° for 1 hr. *Procedure*—The sample (5 g) is extracted (× 4) with a total of 170 ml of water (at 60°), then treated with 10% basic Pb acetate soln. and centrifuged. Excess of Pb is removed with H_2S and the soln. is evaporated to 25 ml; a 5-ml portion is adjusted to pH 5·0 and passed through a column of the resin. Sufficient HCl is added to the eluate to give a concn. of 2 *N* HCl, and the soln. is heated at 120° for 1 hr. The pH is adjusted to 6·2 with aq. NH_3 soln. and the product is submitted to bio-assay, giving the asparagine. The column is eluted with 0·1 *N* HCl and a similar bio-assay is made on the eluate for aspartic acid.

III. Determination of theanine by microbiological assay. Zenji Nagashima and Muneyuki Nakagawa. *Ibid.*, 1956, **30** (10), 637–640.—The bio-assay of theanine (N-ethyl-γ-glutamine) (**III**), one of the flavour-regulating components of tea leaf, was studied with *Lactobacillus arabinosus* (**IV**). **III** is converted into glutamic acid (**V**) by heating with 2 *N* HCl at 120° for 1 hr. since **III** is inactive towards **IV**. The presence of α-oxoglutaric acid, glutamine and glutathione vitiates the estimation, but only small amounts of the last two compounds are likely to be present in tea extract. *Procedure*—The sample (5 g) is extracted with water and treated with Pb^{2+} as described above. A portion of the evaporated aq. soln. (25 ml) is adjusted to pH 6·0 and submitted to bio-assay for the determination of free **V**. Another portion is made acid with HCl and heated in a sealed tube; the product is neutralised to pH 6·0 and similarly submitted to bio-assay. The amount of **III** is determined from the difference in the two measurements. K. SAITO

2392. **Determination of added distinctive cations in whisky. V. Spectrophotometric determination of molybdenum and nickel.** M. J. Pro and R. A. Nelson (Alcohol and Tobacco Tax Div. Lab., Internal Revenue Service, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1956, **39** (4), 945–951.—In the determination of Mo, the organic matter in the whisky is destroyed by heating with H_2SO_4, HNO_3 and $HClO_4$, and the prepared soln., acidified with HCl, is treated with KSCN. The liquid is shaken with *iso*amyl alcohol to extract the Mo^V - thiocyanate complex. After dilution of the soln. to known volume, the extinction is read at 465 mμ and the content of Mo is read from a linear calibration curve prepared by using whisky containing microgram quantities of Mo. For the determination of Ni, H_2SO_4 - HNO_3 is used to destroy the organic matter, the residue after fuming being ignited at 500°, dissolved in water, and treated with Br and conc. aq. NH_3 soln., followed by dimethylglyoxime. After dilution, the extinction is measured at 450 mμ and the content of Ni is read on a calibration curve. None of the ions tested interfered individually with the determination of Mo when present in quantities up to 10 p.p.m., or with the determination of Ni if present at 1 p.p.m., but Cu, Cr, Mn and Al interfered at 5 p.p.m.

A. A. ELDRIDGE

2393. **Determination of distinctive added organic materials in whisky. VI. Spectrophotometric determination of citric acid.** M. J. Pro and R. A. Nelson (Alcohol and Tobacco Tax Div. Lab., Internal Revenue Service, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1956, **39** (4), 952–957.—Whisky (10 ml) is evaporated to a vol. of 2 to 3 ml, then diluted with H_2O to 25 ml and centrifuged. An aliquot of the supernatant liquid is treated at 15° with H_2SO_4 and HPO_3, followed by KBr solution (24%, w/v) (0·50 ml) and $KMnO_4$ (saturated solution, 1·50 ml). Excess of $KMnO_4$ is removed with H_2O_2, and the liquid is shaken with heptane. An aliquot of the heptane phase is added to a mixture of KOH solution and pyridine, and heated, cooled, and centrifuged. An aliquot of the pyridine layer is added to ethanol and the extinction read at 535 mμ. Standards of citric acid are similarly treated. A small blank is attributed to homologues of citric acid frequently present in whisky. The method is accurate, rapid and specific. For samples containing 10 p.p.m. of citric acid, 9·5 to 10·2 p.p.m. were reported.

A. A. ELDRIDGE

2394. **Application and analysis of synthetic carotenoids in biological material and foodstuffs.** O. Isler, S. Nobile and P. Zeller (Hoffmann-La Roche & Co. A.-G., Basel). *Mitt. Lebensmitt. Hyg., Bern*, 1956, **47** (5), 422–423.—The extinction coefficients of zeaxanthin, kryptoxanthin and β-carotene are too similar for adequate analysis of these complexes. Chromatography on Al_2O_3, with light petroleum as solvent and ether as eluent, was successfully used.

D. P. FELIX

2395. **Determination of small amounts of monobromoacetic acid and monobromoacetic acid esters.** B. Hansen (Copenhagen Univ., Denmark). *Acta Pharm. Tox., Kbh.*, 1956, **12** (4), 389–398.—The microbiological determination of bromoacetic acid (**I**) described previously (*Ibid.*, 1956, **12**, 379) is adapted for use in the estimation of the esters of **I** in fruit juices and jams. The method will determine 4 to 5 mg of ester per litre with an accuracy of ±20%, provided that other ether-soluble preservatives are absent. The chemical method described requires preliminary ether extraction of the sample, diluted if necessary, and adjusted to pH 1 to 2 with H_2SO_4. The ether extract is treated with an excess of aq. KOH and MgO, then evaporated to dryness, and the residue is heated and ignited at <500°. The ash is dissolved in dilute H_2SO_4 and diluted to known vol. Aliquots are then tested for Cl^-, Br^- and I^-, and Br^- are determined by oxidation with $K_2Cr_2O_7$ in dilute H_2SO_4 in a Conway micro-diffusion apparatus. The Br is absorbed in KI soln. and the liberated I is determined by titration with 0·01 *N* $Na_2S_2O_3$. The method is sensitive to 0·5 mg of ether-soluble Br per litre and gives a recovery of 94%. Modifications are given for determining the chlorine and iodine analogues of **I**.

W. H. C. SHAW

2396. **Isolation and detection of 3-aminobutyric acid in fruit juice.** Sakae Shinano, Toshio Kaya and Hiroshi Kaneko (Dept. Agric. Chem., Fac. Hortic., Chiba Univ., Matsudo, Chiba Prefecture). *J. Agric. Chem. Soc., Japan*, 1956, **30** (3), 175–178.—The isolation and detection of 3-aminobutyric acid (**I**) in grape and tomato juice was studied by ion exchange followed by paper chromatography. The juice (2 to 10 litres) was mixed with an equal vol. of ethanol, filtered and condensed to one-quarter of the vol. The product was added to a column of Diaion BK (H) (phenolsulphonic acid resin), which was eluted with 0·15 *N* aq. NH_3 soln. The later fraction, containing **I**, was successively treated with activated charcoal and Amberlite IRC-50 to remove tyrosine and histidine, then evaporated to a syrupy liquid and mixed with absolute ethanol to precipitate **I**, which is recrystallised from aq. ethanol as long prisms.

K. SAITO

2397. **Separation and identification of fatty acids. XIX. Paper-chromatographic analysis of fatty acids in natural fat.** Manjiro Noda, Osamu Hirayama and Yoshiyuki Inouye (Biochem. Lab., Coll. Agric., Kyoto Univ., Sakyo-ku, Kyoto). *J. Agric. Chem. Soc. Japan*, 1956, **30** (2), 106–111.—The paper-chromatographic separation of unsaturated fatty acids and higher saturated fatty acids was studied by the use of addition compounds of their methyl esters with Hg acetate, by the hydroxamic acid method (reversed-phase chromatography) and by the 2:4-dinitrophenylhydrazide method. The treated sample is spotted on filter-paper impregnated with tetralin and developed with a mixture of 90% methanol, acetic acid and tetralin (10 + 2 + 1). The results were applied to the analysis of fatty acids in cows' milk fat. The sample is saponified, extracted with ether and fractionated into 10 groups by distillation, for subsequent chromatographic examination. The first method is the best for unsaturated compounds and the last for saturated compounds.

K. SAITO

2398. **Analysis of mixtures of higher saturated normal fatty acids: a comparison of reversed-phase partition chromatography and ester fractionation.** G. A. Garton and A. K. Lough (Rowett Res. Inst., Bucksburn, Aberdeenshire, Scotland). *Biochim. Biophys. Acta*, 1957, **23** (1), 192–195.—The reversed-phase partition chromatographic method of Howard and Martin (*Brit. Abstr. C*, 1950, 487) for higher fatty-acid analysis is extended to include the odd-numbered, normal fatty acids C_9 to C_{19}, inclusive. The method is applied to the analysis of the saturated non-volatile fatty acids from four samples of

bovine milk fat, and the results agree well with those obtained by the ester-fractionation procedure. Individual ester fractions are examined by the method given, and the significance of the results is discussed. W. H. C. SHAW

2399. **Rapid spectrophotometric method for determining the linoleic and linolenic acid components of soya-bean oil.** F. I. Collins and V. E. Sedgwick (U.S. Dept. of Agric., Urbana, Ill., U.S.A.). *J. Amer. Oil Chem. Soc.*, 1956, **33** (4), 149–152.—Soya-bean oil can be satisfactorily isomerised by heating at 180° with 11% KOH - glycerol in open bottles in a forced-draught air-oven, and thus a rapid means of analysing soya-bean oil for linolenic and linoleic acids has been developed. The extinctions of methanol soln. of the isomerised oil were measured at 268 mμ and 233 mμ, in special cells with quartz windows and short light paths; the percentages were calculated by a nomogram. Details of methods and apparatus are given and tables illustrating the reproducibility of analyses of soya-bean oil for linolenic and linoleic acids are shown. P. HAAS

2400. **Recording instrument for measuring oxygen absorption [of lipids].** E. B. Lancaster, E. D. Bitner and R. E. Beal (Northern Utilization Res. Branch, Peoria, Ill., U.S.A.). *J. Amer. Oil Chem. Soc.*, 1956, **33** (1), 36–38.—Two simple and inexpensive electrical circuits are described which translate change in mercury level in a manometer into an e.m.f. of sufficient magnitude for application to a standard d.c. potentiometric recorder. When the recorder is coupled to a simple closed system, containing a small quantity of lipid material and O or air, it can be adapted to record continuously the absorption of O by the oil or fat. The breakpoint, which is a measure of the stability of the sample, can be obtained from the resulting curve. The apparatus has been employed successfully to determine the oxidative stability of hydrogenated soya-bean oil. The recording manometer should be useful also for measuring and recording pressure changes in other systems or for use in the quality control of edible oils, etc. P. HAAS

2401. **Determination of antioxidants in edible fats.** C. Anglin, J. H. Mahon and R. A. Chapman (Food and Drug Directorate, Dep. Nat. Health and Welfare, Ottawa, Canada). *J. Agric. Food Chem.*, 1956, **4** (12), 1018–1022.—Methods are described for determining butylated hydroxytoluene (BHT), butylated hydroxyanisole (BHA), propyl gallate (PG), nordihydroguaiaretic acid (NDGA) and all combinations except those containing both PG and NDGA. BHA and BHT are separated from the fat and other antioxidants by distillation with superheated steam. The distillate is analysed for the sum of BHA and BHT with $FeCl_3$ - 2:2′-dipyridyl reagent, and for BHA with 2:6-dichloro-*p*-benzoquinonechlorimine reagent; BHT is determined by difference. PG and NDGA are extracted from a CCl_4 soln. of the fat with ethanol (50%) and are determined with $FeSO_4$ soln. buffered to an appropriate pH. S.C.I. ABSTR.

2402. **Polarographic estimation of vitamin A in cod-liver oil.** W. Keller and F. Weiss (Pharm. Inst. der Humboldt Univ., Berlin). *Z. anal. Chem.*, 1957, **154** (4), 241–245.—The application of an earlier method (*cf. Anal. Abstr.*, 1956, **3**, 1524) to cod-liver oil is reported. The preparation of the sample for polarography involves saponification and ether extraction for vitamin-A concentrates and further, chromatographic, purification for cod-liver oil. Heat the sample (up to 1 g, containing 100 to 300 μg of vitamin A) with ethanolic KOH (2 *N*) (5 ml) for 15 min. on a boiling-water bath under reflux. Add H_2O (30 ml) to the hot product and extract with ether (3 × 30 ml). Wash the extract with H_2O (15 ml), aq. KOH (0·5 *N*) (15 ml) and then H_2O until it is neutral to phenolphthalein. Dry with Na_2SO_4 (5 g). Distil off the ether *in vacuo* with a slow stream of N on a water bath at 30°. For cod-liver oil, cover a 10-cm column of alumina with light petroleum (boiling range 90° to 100°), add the residue (from the ether extract) dissolved in light petroleum (10 ml), and develop with light petroleum (30 ml). Change the receiver and elute with a mixture of ether (7·5 ml) and light petroleum (42·5 ml). Vitamin A shows a yellow - green fluorescence in u.v. Evaporate the eluate *in vacuo*, dissolve the residue in methanol and estimate polarographically. The estimation must be carried out rapidly to minimise oxidation. Sets of polarograms are shown. Results are in good agreement with those of colorimetric and biological methods. G. BURGER

2403. **Critical observations on the colorimetric determination of organic substances under polarised conditions, with special reference to biological material. II. [Vitamin A].** W. Krauss, H. Caspers and F. Käsbauer (Inst. of Physiol. u. Ernährung der Tiere, Munich, Germany). *Z. anal. Chem.*, 1957, **154** (5), 333–340.—The Carr - Price reaction for the determination of vitamin A is discussed. Several possible causes of error are established by a mathematical treatment. Errors arise because the amount of reagent used is not always present in sufficient concn. to shift the equilibrium of the reaction completely in the direction of the polarised complex to be measured. Owing to variation of the dissociation constant of the complex under differing conditions this is not always true. Certain general precautions are suggested by which the errors are minimised but not completely obviated. The method is not advised for routine analysis, since it requires a highly trained operator with considerable experience in this field. L. S. ADLER

2404. **Special apparatus for the serial determination of vitamin B_1 by means of column chromatography.** I. Antener (Vitaminlabor Afico S.A., La Tour-de-Peilz). *Mitt. Lebensmitt. Hyg., Bern*, 1956, **47** (5), 415–421.—An apparatus is described for the simultaneous determination of several vitamin-B_1 soln. by chromatography under vacuum on Decalso. After passage of the sample in the cold, the column is washed with water at 100° before elution with acidified KCl. Determination of vitamin B_1 by two microbiological methods (*Lactobacillus fermentum* and *Phycomyces blakesleeanus*) and a physicochemical method (thiochrome test) before and after chromatography shows this purification to be necessary for all three methods. D. P. FELIX

2405. **Determination of vitamin B_2 [riboflavine] in multivitamin preparations.** Keiji Inami and Akihide Ohara (Takamine Lab., Sankyo Co. Ltd., Shinagawa-ku, Tokyo). *Ann. Rep. Takamine Lab.*, 1955, **7**, 118–122.—The determination of riboflavine in partly deteriorated multivitamin preparations was studied by the riboflavine fluorimetric

and the lumiflavine methods. The former method provides satisfactory results in the absence of fat-soluble vitamins. For their removal, extraction with $CHCl_3$ suffices. When the sample has been subject to autoxidation, appreciable luminescence (due to an unknown substance) remains in the aq. layer; the analysis should then be carried out by the latter method. K. SAITO

2406. **Factors influencing validity and confidence limits of pantothenic acid estimation. Microbiological assay with *Lactobacillus casei*.** M. F. Clarke (The Woman's Med. College of Pennsylvania, Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 135–139.—In the microbiological assay of pantothenate with *Lb. casei* in peptone media there is a linear relationship between the logarithm of pantothenate per tube and the amount of acid formed upon incubation. The use of an enzyme-hydrolysed casein medium in place of a peptone medium results in a straight line of much greater slope. Factors influencing this slope were examined and the most favourable conditions for assay occurred when 3% glucose and heavier inoculum were employed and when dosage corresponded to 0·125 to 0·25 μg of Ca pantothenate. G. P. COOK

2407. **Determination of tocopherol in autoxidising methyl esters of fatty acids.** H. J. Lips (Div. Appl. Biol., Nat. Res. Lab., Ottawa, Canada). *J. Amer. Oil Chem. Soc.*, 1956, **33** (9), 426–428.—Autoxidising methyl esters of fatty acids interfere with the determination of α-tocopherol by u.v. spectrophotometry (*cf.* Cuthbertson *et al.*, *Biochem. J.*, 1940, **34**, 34) or by the dipyridyl colorimetric method (*cf.* Kaunitz and Beaver, *J. Biol. Chem.*, 1944, **156**, 653, 661). Interference with the colorimetric method is removed by H_2SO_4 treatment, but spectrophotometry is applicable only when the tocopherol is completely separated from the oxidised fat. Separation is not possible with H_2SO_4, but is possible by room-temp. saponification in an ethanol - light petroleum system protected by pyrogallol. The sensitivity of the spectrophotometric method is increased by oxidising tocopherol quantitatively to the *p*-quinone with 2 *N* HNO_3. I. JONES

2408. **Identification of stored-products insects by the microbiology of the exoskeleton. III. Identification of larval fragments and their significance in sanitation analyses.** O'D. L. Kurtz (Div. Microbiol., Food and Drug Admin., Dept. Health, Educ. and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1956, **39** (4), 990–1014.—Morphological characteristics of larval fragments useful in identification of various insects that may be found in stored products are described and illustrated. A. A. ELDRIDGE

See also Abstracts 2234, 2235, 2308.

Sanitation

2409. **New colorimetric method for estimating carbon monoxide in air.** G. Ciuhandu (Lab. Toxicol., Inst. Igienă, Timişoara, Romania). *Stud. Cercet. Chim., Bucharest*, 1955, **3** (3–4), 243–251.—Carbon monoxide is estimated colorimetrically as the coloured silver sol that is obtained when an alkaline soln. of Na *p*-sulphamylbenzoate reacts with the CO in the presence of Ag^+. After allowing the reagent to stand in contact with the sample of air for 22 to 24 hr., the extinction of the soln. is measured at 510 mμ. By this method, 0·001 to 0·2% of CO in air can be determined. J. H. WATON

2410. **Colorimetric determination of pyridine in air.** M. I. Tarenko (Inst. of the Hygiene of Work and Occupational Diseases, Ministry of Health, Gruz S.S.R.). *Zhur. Anal. Khim.*, 1956, **11** (5), 644–645.—The reaction of pyridine with thiocyanogen bromide and aniline (Shmuk and Borozdina, *Zhur. Prikl. Khim.*, 1940, **13**, 776) is used. The sample of air is passed at a rate of 20 to 25 litres per hr. through a vessel containing 2 ml of 0·1 *N* H_2SO_4. The soln. is neutralised and treated with 0·5 ml of a soln. prepared by adding 5% NH_4SCN soln. dropwise to bromine water until the latter is decolorised, and then with 1 ml of water saturated with freshly distilled aniline. The colour is compared after 10 to 15 min. with a scale of standards. A new colour reaction, although of low sensitivity for pyridine bases, is also described. When an aq. soln. of pyridine is saturated with H_2S, and further treated with 30 to 35% NaOH soln., a green coloration appears. G. S. SMITH

2411. **Practical use of the electrochemical method of determination of oxygen in water. I. Principles: long-term measurements.** H. Ambühl. *Schweiz. Z. Hydrol.*, 1955, **17**, 123–155.—The basic principles of electrochemical determination of O and the problems that arise in the practical application of the method are discussed. N. E.

2412. **Flame-photometric determination of the contents of sodium and other alkali elements in industrial waters and catalysts.** N. Zaĭdman and D. Orechkin. *Nov. Neft. Tekh.*, 1955, **5**, 25–30; *Ref. Zhur., Khim.*, 1956, Abstr. No. 54,735.—Flame-photometric apparatus calibrated to cover the concn. range 0·005 to 5% of Na_2O is described. In industrial waters, contents of $Na_2O \geqslant 0{\cdot}05\%$ can be determined fairly accurately in the presence of $FeCl_3$ and $CrCl_3$, but $CuCl_2$ lowers the results. The method is applicable to the determination of Na in alumina, and the results agree with those obtained in the normal way, which takes 30 times as long. Potassium ($\leqslant 0{\cdot}001\%$) can also be determined. Rapid determinations of Na and other elements in certain catalysts and intermediate products can be carried out. G. S. SMITH

2413. **Microgasometric determination of dissolved oxygen and nitrogen.** P. F. Scholander, L. Van Dam, C. L. Claff and J. W. Kanwisher (Marine Biol. Lab., Woods Hole, Mass., U.S.A.). *Biol. Bull.*, 1955, **109**, 328–334.—The gases are liberated from the polluted water or biological fluid in a special apparatus by the generation of CO_2 from citric acid and K_2CO_3. The CO_2 is removed by absorption by KOH soln. and the O and N are determined in the bubble by known methods. N. E.

2414. **Colorimetric determination of small amounts of phenol in water by means of 2:6-dichloro-*p*-benzoquinonechlorimine.** E. A. Bozhevol'nov. *Trudy Vses. Nauch. Inst. Khim. Reaktivov*, 1956, (21), 38–42; *Ref. Zhur., Khim.*, 1956, Abstr. No. 54,848.—By interaction of Na phenoxide with 2:6-dichloro-*p*-quinonechlorimine (**I**) there is formed Na 2:6-dichlorophenolindophenoxide, which is intensely blue in alkaline soln. The sample of

water (250 ml) is treated with 2·5 g of $Na_2B_4O_7$, to give a pH of 9·24, and 1·5 ml of a 0·25% soln. of **I** in ethanol, and examined after 2 hr. on a Hilger photo-electric colorimeter with a red filter. The reaction is sensitive to 10^{-6} %. The method can be made more sensitive (10^{-9} %) by extracting the coloured complex with 25 ml of *iso*amyl alcohol free from furfuraldehyde. The reaction is more specific than that with diazotised sulphanilic acid; 1- and 2-naphthol, quinol, pyrogallol, pyridine and *p*-cresol do not interfere. G. S. SMITH

2415. **Mass-spectrometric determination of volatile solvents in industrial waste water.** G. P. Happ, D. W. Stewart and H. C. Cooper (Eastman Kodak Co., Rochester, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (1), 68–71.—A variety of volatile organic compounds dissolved in water at the p.p.m. level can be identified and determined with the mass spectrometer. Interference from dissolved or suspended solids is eliminated by sampling the vapour phase in equilibrium with the liquid in an air-free system. A continuous-flow device is also described for the sampling of sewage waters. A recovery of 95% was obtained from sewage containing 100 p.p.m. of added dichloromethane. G. P. COOK

2416. **Papilloma of the bladder in the chemical industry. Analytical methods for the determination of benzidine and 2-naphthylamine, recommended by A.B.C.M. Sub-committee.** L. T. Butt and N. Strafford (I.C.I. Ltd., Dyestuffs Div., Blackley, Manchester). *J. Appl. Chem.*, 1956, **6** (12), 525–539.—Samples are taken of a measured volume of air, dust deposits from air, clothing, and chemicals suspected of contamination with benzidine or 2-naphthylamine. Benzidine is estimated colorimetrically by the chloramine-T oxidation reaction, or, in the presence of related amines, by the 1:2-naphthaquinone-4-sulphonate chromatographic method. In the absence of other amines, benzidine or 2-naphthylamine is determined by the 3-hydroxy-2-naphthoic acid coupling method. 2-Naphthylamine is estimated by the hypochlorite reaction without interference from primary aromatic amines and, in admixture with any other amine, by coupling with R-salt (2-hydroxynaphthalene-3:6-disulphonic acid) and chromatographing. F. J. M.

See also Abstracts 2368, 2467.

Agriculture and Plant Biochemistry

2417. **Application of flame spectrophotometry to the analysis of vegetable matter.** M. Pinta and C. Bove (Off. Rech. Sci. et Tech. Outre-Mer, Bondy, France). *Mikrochim. Acta*, 1956, (12), 1788–1817 (in French).—The determination of Na, K, Ca and Mg in vegetable ash is carried out by flame spectrophotometry, with an air - C_2H_2 flame and measurement at the wavelengths 5896/9 A, 6220 A, 7665/99 A and 3810 A, respectively. The mutual interference of the four cations, as well as the interference of Mn, Fe and Al, has been studied, and interference graphs have been constructed from which corrections may be applied. Of the anions, PO_4^{3-} has the greatest influence; the interference of P in the determination of Ca has been particularly studied and techniques have been developed in which correction curves are used or in which PO_4^{3-} are removed by means of ion exchangers. L. A. O'NEILL

2418. **Micro-determination of fluorine in biological material. I. Determination of organic fluorine in plants.** Nobuo Horiuchi (Takamine Lab., Sankyo Co. Ltd., Shinagawa-ku, Tokyo). *Ann. Rep. Takamine Lab*, 1955, **7**, 209–214.—The AOAC method and a new sodium reduction method were compared for the determination of organic fluorine in spray residues (*e.g.*, fluoroacetamide) in plants. The latter method consists in extraction of the plant material with methanol, decomposition with Na in hexanol, extraction of the resulting NaF with water, evaporation, and steam-distillation of HF (as SiF_4) with $HClO_4$ and $AgClO_4$. Neither method provides satisfactory results, presumably owing to volatilisation of organofluorine compounds with methanol. K. SAITO

2419. **A study of the uptake of silicon and phosphorus by wheat plants with radiochemical methods. [Determination of ^{31}Si.]** L. Rothbuhr and F. Scott (Wolverhampton and Staffordshire Tech. Coll., Wolverhampton, England). *Biochem. J.*, 1957, **65** (2), 241–245.—A method is described for the determination of ^{31}Si in biological material with an accuracy of 0·5 μg. The material is dried, weighed, ashed at as low a temp. as possible, and the residue is fused with Na_2CO_3. The resulting mass is dissolved in dil. HCl and the soln. is put into a liquid "counter." The amount of $^{31}SiO_2$ is determined by comparing the counting rates with those of standard soln. J. N. ASHLEY

2420. **Problems and techniques in the analysis of plant material for hemicellulose.** K. T. Williams and A. Bevenue (Agric. Res. Service, U.S. Dept. Agric., Albany, Calif., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1956, **39** (4), 901–918.—Procedures available for the removal of lignin, pectic substances, starch and protein and for the separation and examination of hemicelluloses are reviewed and discussed. For the removal of lignin, treatment with aq. acetic acid and $NaClO_2$ is recommended. An aq. soln. of sodium polymetaphosphate or EDTA (disodium salt) is a good solvent for pectic substances that are not extractable by water. Starch is best removed by enzymic hydrolysis, but no enzyme system capable of completely hydrolysing all starches has yet been found. The effectiveness of proteolytic enzymes also is limited; treatment with a cationic detergent offers the possibility of removal of protein while leaving the hemicelluloses in a condition suitable for examination. Conditions for the isolation of hemicelluloses, which are insoluble in water but soluble in aqueous alkali, are considered, together with their precipitation. Cupriethylenediamine may be used to disperse the mixture of hemicellulose and cellulose, the latter being then pptd. with HCl at controlled pH. The graded hydrolysis of the resulting hemicellulose to determine the nature and mode of association of the monoses is also discussed, and there is a brief examination of the significance of the presence of water-soluble polysaccharides and α-cellulose. (148 references.) A. A. ELDRIDGE

2421. **Simple photometric determination of pentosan in wood.** Yoshio Sakai (Fac. Agric., Tokyo Univ. of Educ., Ikejiri, Setagaya-ku, Tokyo). *J. Agric. Chem. Soc. Japan*, 1956, **30** (5), 256–259.—The colorimetric determination of pentosan (**I**) with benzidine (**II**) (McCance, *Biochem. J.*, 1926, **20**, 1111) was modified for use as a rapid spectrophotometric

procedure. The colour produced between furfuraldehyde (**III**) and **II** reaches max. intensity 20 min. after the addition of the reagent and remains unchanged for 5 min., obeying Beer's law for 0·1 to 0·4 mg of **III** per 5 ml. The conversion of xylose into **III** in 13% HCl was not affected by the presence of H_2SO_4. *Procedure*—The sample (20 mesh) containing 2 to 40 mg of **I** is mixed with 72% H_2SO_4 (5 ml), set aside at 30° for 45 min. with occasional stirring, diluted, filtered and made up to 100 ml. A 1 to 5-ml portion is made up to 5 ml with 0·6 *N* H_2SO_4 and treated with conc. HCl (2·5 ml) for 3·5 hr. in a boiling-water bath. The product is cooled and extracted with benzene (5 ml), and the organic layer is mixed with **II** (0·5 g in a 1 to 1 mixture of absolute ethanol and glacial acetic acid); the extinction is measured at 570 mμ after 20 min., with a mixture of the **II** soln. and benzene (2 + 5) as reference soln. K. SAITO

2422. **Determination of lignin.** H. Brune and K. H. Sieck (Inst. Tierphysiol., Univ. Göttingen, Germany). *Z. Tierernähr.*, 1956, **11**, 253–257.—In the procedure proposed, the residue from the ether extraction in the Weende method for estimating fibre is used. It is boiled in water, centrifuged and washed; digested with pepsin and HCl and washed and digested with H_2SO_4, with control of temp. to not above 23°; the sediment from this digestion is filtered through sand, washed, dried and ignited. The loss is lignin. It is claimed that reproducibility of results is excellent. Heats of combustion of lignin so isolated from a beet product and faeces of wethers fed on the product in question were 6·85 and 6·83 g-cal., so that the method can be used in digestibility experiments. NUTR. ABSTR. REV.

2423. **Spectrophotometric determination of the activity of dehydrogenase in plant-tissue suspensions.** E. S. Mikhallova and G. P. Brin (A. N. Bakh Inst. of Biochem., Acad. Sci., U.S.S.R., Moscow). *Biokhimiya*, 1956, **21** (4), 444–447.—A method based on the decolorisation of Lauth's violet is described. The sample (4 ml) and 1 ml of a 0·1 *M* soln. of a H-donor, *e.g.*, glucose, in a Tunberg tube are kept for 10 min. at 37° to 39° *in vacuo* and then treated with 4 ml of Lauth's violet soln. of suitable concn. The extinction is measured at intervals during 20 to 30 min. G. S. SMITH

2424. **Determination of the exchangeable calcium and magnesium content of the soil by a complexiметric titration method.** J. van der Spek and J. ten Have (Landbouwproefstation Inst., T.N.O., Groningen, Holland). *Chem. Weekbl.*, 1957, **53** (2), 10–14.—Magnesium can be determined directly, by complexiметric titration, in the filtrate obtained after removal of the Ca by pptn., but the usual aq. NH_3 - NH_4Cl buffer must be replaced by a Na_2CO_3 buffer. Alternatively, Ca and Mg can be titrated together, the Ca being subsequently pptd. as oxalate, dissolved in H_2SO_4, and titrated with $KMnO_4$. The soil sample is percolated with 0·5 *N* NaCl and the extract is treated with bromine water made alkaline with aq. NH_3 and boiled. Any slight ppt. is filtered off and the soln. is freed from Ca by the addition of ammonium oxalate and the filtrate is titrated with EDTA after addition of 5% KCN and Na_2CO_3 soln. (80 g of anhyd. salt per litre). For soils containing $CaCO_3$, a correction has to be made for the solubility of this salt in the NaCl extract. P. HAAS

2425. **Determination of free iron oxide in soils.** A. D. Haldane (C.S.I.R.O., Canberra). *Soil Sci.*, 1956, **82**, 483–489.—The sample of sieved (1 mm) soil, not exceeding 4 g nor containing more than 300 mg of Fe_2O_3, is treated with 1 ml of 0·5 *N* NaOH and sufficient H_2O to make a thick paste. The mixture is ground thoroughly and approximately neutralised by 1 ml of 0·5 *N* oxalic acid. Grinding is continued with gradual addition of a buffer soln. containing ammonium oxalate monohydrate (50·5 g) and oxalic acid (18·3 g) in 1 litre (pH, 3·60 to 3·65). When about 40 ml of buffer has been added the mixture is allowed to settle for 1 to 2 min. and the suspension is decanted from the heavier particles into a 100-ml flask. Grinding and decantation are repeated with smaller volumes of buffer until the sample is completely transferred to the flask. After addition of 0·4 to 0·5 g of zinc dust ($Fe_2O_3 < 0·02\%$, passing 300 mesh) and thorough mixing, buffer is added to bring the liquid to the bottom of the neck of the flask. After the soln. has stood for 1 hr., with intermittent shaking, entrained air is removed and the soln. is made up to the mark. An aliquot (20 ml) of the filtrate is digested with H_2SO_4, followed by $HClO_4$, and the contained Fe is reduced with $SnCl_2$ in HCl and finally titrated with 0·042 *N* $K_2Cr_2O_7$. A. G. POLLARD

2426. **Determination of urea in fertilisers and cattle foods.** G. H. Jongen and H. W. Berkhout (Rijkslandbouwproefstation, Maastricht, Netherlands). *Chem. Weekbl.*, 1956, **52** (51), 909–910.—A modification of the method of Watt and Chrisp (*Anal. Abstr.*, 1954, **1**, 1929) is described. The material (1 kg) is shaken with 450 ml of H_2O, 1 g of decolorising carbon and 5 ml of each of soln. (*A*) and (*B*). *Soln. A*—Zn acetate (23·8 g) and glacial acetic acid (3 g) diluted to 100 ml with H_2O. *Soln. B*—$K_4Fe(CN)_6$ (10·6 g) in H_2O to 100 ml. The urea in the filtrate is determined by Watt and Chrisp's method. W. J. WRIGHT

2427. **Assay of acetyl-(*p*-nitrophenyl)sulphanilamide [APNPS] in feeds.** J. W. Cavett (Res. Div., Dr. Salsbury's Lab., Charles City, Iowa, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1956, **39** (4) 964–967.—The feed (5 g) is treated with methanol (80 ml) at 60° for 20 min., then cooled and diluted to 100 ml with methanol; 25 ml of the extract is treated with H_2O (10 ml), 1% $ZnSO_4$ solution (5 ml) and *N* NaOH (3 drops). After 2 min. at 100° the mixture is cooled, diluted to 50 ml and filtered. An aliquot (10 ml) is treated with H_2O (8 ml) and conc. HCl (0·5 ml); after being kept at 100° for 1 hr., the mixture is cooled and diluted to 50 ml. An aliquot (5 ml) is placed in each of 2 colorimeter tubes. To one (for the blank) is added H_2O (1 ml) and coupling reagent (0·5 ml of 0·1% aq. N-1-naphthylethylenediamine dihydrochloride). To the other is added 0·1% $NaNO_2$ solution (0·5 ml), then 0·5% of ammonium sulphamate solution (0·5 ml) and finally coupling solution (0·5 ml). The difference in extinction is read in a photoelectric colorimeter and the APNPS content is read from a standard curve. Arsanilic acid or sulpha drugs cause high results. A. A. ELDRIDGE

2428. **Colorimetric method for the determination of 4-nitrophenylarsonic acid in feed.** J. W. Cavett (Res. Div., Dr. Salsbury's Lab., Charles City, Iowa, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1956, **39** (4), 967–969.—The feed (6 g) is extracted with hot

methanol (80 ml) and after cooling is diluted with methanol to 100 ml. An aliquot (25 ml) is evaporated almost to dryness and heated at 100° (30 min.) with a 1% solution of $Na_2S_2O_4$ in 4% NaOH (50 ml), conc. HCl (1 ml more than produces turbidity) being then added. After being aerated, cooled and diluted to 100 ml, the solution is filtered and aliquots (4 ml) are placed in 2 colorimeter tubes; to each are added 0·5% $NaNO_2$ solution (1 ml) and 2·5% ammonium sulphamate solution (1 ml). To one tube is then added 2 ml of H_2O and to the other 2 ml of a 0·1% aq. solution of N-1-naphthylethylenediamine dihydrochloride. The difference in absorbance is used with standard curves to determine the 4-nitrophenylarsonic acid content. For 20 determinations (0·025% content) the standard deviation was ±0·0012. A. A. ELDRIDGE

2429. **Assay for dinitrodiphenylsulphonylethylenediamine in feed.** J. W. Cavett (Res. Div., Dr. Salsbury's Lab., Charles City, Iowa, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1956, **39** (4), 969–972.—Acetyl-(*p*-nitrophenyl)sulphanilamide (**I**) and dinitrodiphenylsulphonylethylenediamine (**II**) are extracted with hot ethanol. **I** is deacetylated and **II** is reduced by heating with $Na_2S_2O_4$ and NaOH, and the absorbance due to both after they have been treated with $NaNO_2$, ammonium sulphamate and N-1-naphthylethylenediamine is determined in a photo-electric colorimeter. The absorbance due to the quantity of **I** usually present (0·03%) is determined by a similar procedure and deducted. The content of **II** is then read from standard curves. Other sulpha compounds and arsenicals, if present, cause high results. Certain interfering substances, *e.g.*, 4-nitrophenylarsonic acid, can be removed by chromatography. The method is suitable for routine control. A. A. ELDRIDGE

2430. **Polarography of agricultural chemicals. V. Simultaneous determination of copper and zinc in Zinc Bordeaux by polarography.** Shigeru Yamamoto (Takamine Lab., Sankyo Co. Ltd., Shinagawa-ku, Tokyo). *Ann. Rep. Takamine Lab.*, 1955, **7**, 215–220.—The determination of Cu and Zn (≃8%) in Zinc Bordeaux [a suspension containing $CuSO_4$, $Cu(OH)_2$, $ZnSO_4$, $Zn(OH)_2$ and $Ca(OH)_2$] was studied in aq. NH_3 soln. containing NH_4Cl. A typical polarogram was obtained which enabled a simultaneous determination to be made. The sample soln. (≃0·3 g) is dissolved in 10% H_2SO_4 (10 ml) and diluted to 100 ml; a small portion (≃1 ml) is made up to 10 ml with 0·5% gelatin soln. (0·2 ml) and ammonium buffer (13·37 g of NH_4Cl and 17 ml of 28% aq. NH_3 soln. in 250 ml) and submitted to polarography ($E_{\frac{1}{2}} = -0·5$ V *vs.* the S.C.E. for Cu and −1·36 V for Zn).
K. SAITO

2431. **Chemical aspects of pyrethrum analysis. II.** R. G. Barradas. *Chem. Age*, 1957, **77**, 17–20.—A review of the more modern aspects of pyrethrum assay is continued. The chemistry of allyrethronyl chrysanthemate (allethrin) is discussed in relation to its determination in insecticides. H. B. HEATH

2432. **Isolation of piperonyl butoxide from oils, fats and waxes.** H. L. Williams and J. P. Sweeney (Stored-Product Insects Lab., Savannah, Ga., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1956, **39** (4), 975–980.—In the chromatographic procedure described, Florisil is used as adsorbent, and mixtures of Skellysolve F with ethyl ether as eluting solutions. Colour is developed with tannic acid dissolved in a mixture of acetic and phosphoric acids, and the extinction of the blue solution is read at 625 mμ. Recoveries of 92·6 to 106·6% of piperonyl butoxide (a synergist for pyrethrum) added to various grains, nuts and paper are reported. A. A. ELDRIDGE

2433. **Note on the analysis of commercial insecticide dusts containing two chlorinated hydrocarbons.** D. P. Johnson (Anal. Div., N. Carolina Dept. Agric., Raleigh, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1956, **39** (4), 1017.—If the material, which contains any two known chlorinated hydrocarbons having different percentages of Cl, is free from other ether-soluble and chlorine-bearing substances, a reasonably accurate estimate of the amount of each constituent may be made by determining the total Cl content and the ether-soluble extract. A. A. ELDRIDGE

2434. **The estimation of benzene hexachloride [hexachlorocyclohexane] in presence of pentachlorocyclohexene.** F. R. Bradbury and H. Standen (Res. Dept., I.C.I. Ltd., Widnes). *Chem. & Ind.*, 1957, (5), 140.—The specificity of the method of Armstrong *et al.* (*Ann. Appl. Biol.*, 1951, **38**, 555) for the determination of small quantities of γ-hexachlorocyclohexane (**I**) in the presence of pentachlorocyclohexene (**II**) has been established. The reading for 100 μg of **II** treated by this method was not significantly different from the reagent blank reading. **II** is converted into tetrachloroadipic acid in the first nitration and remains in the aq. phase when it is made alkaline and **I** is extracted with CCl_4 - ether. G. S. ROBERTS

2435. **Separation and identification of chlorinated organic pesticides by paper chromatography. VIII. Technical DDT, *p,p'*-DDT, DDA, DDD, DDE, 4:4'-dichlorobenzophenone and 2:4'-dichlorobenzophenone.** L. C. Mitchell (Div. Food, Food and Drug Admin., Dept. Health, Educ., Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1956, **39** (4), 980–985.—Four sets of spotted papers are prepared; three sets are sprayed with soya-bean oil in ether (1 diluted to 200) and developed respectively with aq. acetone, aq. 2-methoxyethanol, and aq. pyridine. The fourth set is sprayed with dimethylcyanamide in ether (20 diluted to 100) and developed with 2:2:4-trimethylpentane. When dry, the papers are sprayed with a reagent prepared by dissolving $AgNO_3$ (1·7 g) in water, adding 2-phenoxyethanol (10 ml) and ethanol (50 ml) and diluting to 200 ml with H_2O. After air-drying, the papers are exposed to strong u.v. light. Six of the seven pesticides are identified by the order of separation and distance from one another. The chromogenic reagent reveals as little as 1 to 3 μg.

IX. Aldrin, DDE, dieldrin, DDT, lindane, methoxychlor, Perthane [1:1:1-trichloro-2:2-di-(*p*-ethylphenyl)ethane] and Rhothane (DDD). L. C. Mitchell. *Ibid.*, 1956, **39** (4), 985–990.—Two sets of duplicate spotted papers are prepared. One set is sprayed with dimethylcyanamide in ether (20 ml diluted to 100 ml) and developed with 2:2:4-trimethylpentane or colourless mineral oil, $d^{15·5}$ 0·775 to 0·825. The other set is sprayed with soya-bean oil in ether (1 ml diluted to 100 ml or 200 ml) and developed with aq. methanol (15 ml of H_2O diluted with methanol to 100 ml). When dry, the papers are sprayed with the chromogenic agent prepared as described in Part VIII, above. After evaporation of the water, the papers are exposed to strong u.v. light. A. A. ELDRIDGE

2436. **Determination of *p*-chlorophenyl *p*-chlorobenzenesulphonate in Sappiran and its emulsion.** Kiyoharu Okada and Tetsuo Uchida (Takamine Lab., Sankyo Co. Ltd., Shinagawa-ku, Tokyo). *Ann. Rep. Takamine Lab.*, 1955, **7**, 220–225.—Kutschinski's colorimetric method (*Brit. Abstr. C*, 1952, 569) for the determination of *p*-chlorophenyl *p*-chlorobenzenesulphonate (**I**) was modified to minimise the deviation and applied to the determination of Sappiran, a commercial product of **I**, and its 25% emulsion. *p*-Chlorophenol, obtained by the alkaline hydrolysis of **I**, produces a red colour with 4-aminophenazone (**II**) and $K_3Fe(CN)_6$ (**III**), which is stabilised by the addition of a borax buffer (pH 9·3). The sample (≃250 mg) is diluted to 50 ml and a portion is refluxed with 0·2 *N* ethanolic KOH (10 ml) for 15 min. The product is diluted to 100 ml with 0·025% Na_2CO_3 soln. and a 2-ml portion is made up to 25 ml with 0·5% **II** (0·5 ml), 8% **III** (0·25 ml) and Kolthoff's borax buffer (pH 9·3). The extinction is measured at 510 mμ after 20 min. To eliminate the interference from phenolic compounds, including *m*-cresol in the Sappiran emulsion, the extinction of the diluted sample soln. is deducted from the measured value.
K. SAITO

2437. **Bromimetric determination of monochlorophenoxyacetic acids in 2:4-D preparations.** A. N. Shebuyeva, A. I. Peshekhonova, K. G. Kirilenko and N. K. Kurcheninova. *Khim. Prom.*, 1955, **7**, 46–47; *Ref. Zhur., Khim.*, 1956, Abstr. No. 47,368.—The method is based on reaction with Br under conditions in which di- and tri-chloro-substituted phenoxyacetic acids are not brominated. To 3 to 3·5 g of 2:4-D (dried at 70° to 80°), or 4 to 4·5 g of a 2:4-D preparation, add 150 ml of water and 10 to 15 % NaOH till alkaline, and warm till dissolved. Cool the soln. to between 20° and 25° and make up to 250 ml. To 100 ml add 0·1 *N* KBr - $KBrO_3$ (50 ml) and HCl (10 ml) and set aside in the dark for 10 min. at 20° to 25°. Then stand the flask for 10 min. in ice water, add a 20% soln. of KI (10 ml), and titrate the liberated iodine with 0·1 *N* $Na_2S_2O_3$.
C. D. KOPKIN

2438. **Determination of captan.** J. Wagner, V. Wallace and J. M. Lawrence (Dept. Agric. Chem., State Coll. of Washington, Pullman, U.S.A.). *J. Agric. Food Chem.*, 1956, **4** (12), 1035–1038.—The method is based on the characteristic colour formation given by captan (N-trichloromethylthio-4-*cyclo*hexene-1:2-carboxyimide) with resorcinol in alkaline medium. Addition of $Na_2S_2O_4$ minimises interference from air oxidation. The method is most useful in the range 3 to 30 μg, but a semi-quant. measure of 0·4 μg can be obtained.
S.C.I. ABSTR.

2439. **Reactions of barbiturates in the determination of OO-diethyl O-*p*-nitrophenyl thiophosphate according to Averell and Norris.** W. Paulus and H. J. Mallach (Inst. Gerichtl. Medizin, Univ., Bonn). *Arzneimittel-Forsch.*, 1957, **7** (1), 72–74.—In the determination of OO-diethyl O-*p*-nitrophenyl thiophosphate (E605) (parathion) (**I**), 8 out of 10 barbiturates (although containing no cyclic nitro or amino group) gave a positive Averell and Norris reaction (*cf. Brit. Abstr. C*, 1949, 170). The carbamide grouping seems to be important for a positive reaction. Exceptions were 5-*sec.*-butyl-5-(2-methylthioethyl)thiobarbituric acid and cyclobarbitone. The colour was determined spectrophotometrically (540 to 560 mμ) but it was difficult to distinguish between azo dyes and **I**. Fairly large amounts of barbiturates (90 mg) were needed to give the reaction. Steam-distillation in acid, alkaline, and neutral media failed to split the **I** - barbiturate complex quantitatively, and it is suggested that column chromatography be used for the effective separation of Averell-positive compounds.
D. P. FELIX

See also Abstracts 2300, 2375.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

2440. **The accuracy of glass micro-volumetric apparatus and the preparation of solutions on the micro scale.** G. Gorbach and A. Haack (Inst. Biochem. Tech. and Nutrition, Tech. High School, Graz, Austria). *Mikrochim. Acta*, 1956, (12), 1751–1756 (in German).—Commercial volumetric flasks are unsuitable for measurement of small volumes as errors of ±0·4% can arise, but Pregl wash-out pipettes are suitable. Self-levelling micro-pipettes which are capable of an accuracy >±3 μl are easier and quicker to use. These are described. They may be used with all liquids with a sp.gr. ≃1. The use of Pregl pipettes for preparing soln. of known concn. is described.
P. S. STROSS

2441. **Preparation of hydrofluoric acid free from heavy metals and silicon in a polyethylene apparatus.** H. Stegemann (Med. Forschungsanstalt Max Planck Ges., Göttingen). *Z. anal. Chem.*, 1957, **154** (4), 267–269.—The apparatus described consists of a train of bottles constructed entirely from commercially available polyethylene parts. The HF is purified by passage through a packing of polyethylene beads and a suspension of NaF in water, and absorbed in water. The water used is distilled water further purified by distillation in a stream of N in a polyethylene apparatus (described). The product is 50% HF containing < 1 μg of Cu or Fe, and< 5 μg of Si, per ml.
G. BURGER

2442. **Two simple micro-filters.** J. T. Stock and M. A. Fill (Norwood Tech. Coll., London). *Lab. Practice*, 1957, **6** (1), 38–39.—The first micro-filter combines the extreme simplicity of the Schwinger device with the rigidity of one described by the authors (*Anal. Abstr.*, 1954, **1**, 2006). When there is more than about 30 mg of product, a second form is described which is a modification of the Willstätter "nail" device.
O. M. WHITTON

2443. **A new automatic apparatus for the determination of ten-milligram quantities of carbon and hydrogen in organic substances.** B. Bobrański (Pharm. Inst. of Medical Acad., Wrocław, Poland). *Mikrochim. Acta*, 1956, (12), 1735–1746 (in German).—The variation of pressure in the combustion tube produced by the combustion of the sample is used for automatically regulating the rate of heating. The micro-boat containing the sample is heated by high-frequency induction. The current in the high-frequency generator is controlled by a manometer. The combustion proceeds under constant pressure and therefore in an excess of oxygen. Diagrams of the combustion-tube train, oxygen-pressure regulator, manometer, H.F. electrodes and

circuit of the oscillator are given. Oxides of nitrogen are absorbed in PbO_2 at 190°, water in anhydrous $CoCl_2$, and CO_2 in ascarite. The oxygen flow-rate is 8 ml per min. If the sample contains more than 10% of N, a tube containing MnO_2 is placed between the $CoCl_2$ and the ascarite. P. S. STROSS

2444. **Simple apparatus for the rapid study of paper partition chromatography.** Huo-Ping Pan (M.I.T., Cambridge, Mass., U.S.A.). *Arch. Biochem. Biophys.*, 1956, **64** (2), 311–314.—The simple inexpensive apparatus described is suitable for the rapid selection of suitable solvent mixtures for the paper partition or adsorption chromatography of a particular sample by the ascending technique. A test-tube (1 cm × 7·5 cm) is sealed at its base to another test-tube (5 cm × 40 cm) about 2 cm from the bottom to form a vertical side-arm, which is closed with a cork. A strip of filter-paper (4 cm × 35 cm) is spotted with the sample about 3 cm from the lower end and supported in the larger tube from a cork fitted with a wire loop. A test-tube (1·5 cm × 12·5 cm) containing the stationary phase is supported in the tube by a wire trapped between the cork and the mouth of the tube. After suitable equilibration, sufficient of the mobile phase is introduced through the vertical side-arm to start development. W. H. C. SHAW

2445. **Improvements in or relating to [coaxial cylinder] viscometers.** E. W. Merrill. Brit. Pat. 763,875; date appl. 3.1.55. U.S.A. date appl. 25.1.54.—The viscometer comprises two rotatable, opposed, linearly co-extensive, coaxial cylindrical surfaces having a difference in radius which is only a minor proportion (>1%) of the radius of either. The alignment of these surfaces is accurately maintained by a radial anti-friction bearing, and the liquid under test is injected directly into the annular space between the surfaces by means of a syringe. The inner cylinder is rotated and the force required to constrain the outer surface against rotation is measured, *e.g.*, by means of a spring dynamometer or a laboratory beam-balance. The instrument measures the parameters of viscosity, *i.e.*, shearing stress and velocity gradient, directly, and does not require the use of any correction factors except a readily measured instrument factor. J. M. JACOBS

2446. **Measurement of osmotic pressure [of aqueous] colloidal [solutions].** National Research Development Corp. (Inventor: D. S. Rowe). Brit. Pat. 748,463; date appl. 24.9.53.—An osmometer comprises two upright, tubular columns. One for containing the colloidal solution is adapted so as to be connected at its open upper end to a source of pressure and at its lower end to be open to one side of a semi-permeable membrane. The other column, for containing the solvent, is open to ambient pressure at its upper end and is connected through a trap to a compartment closed by a diaphragm (of rhodium-plated brass foil) and exposed to the side of the membrane opposite to the column for the colloidal solution. The diaphragm is connected to a sensitive means for detecting small deflections, which includes a mechano-electronic transducer (*e.g.*, a valve with a movable anode).
J. M. JACOBS

See also Abstract 2246.

Optical

2447. **Photo-electric methods for the estimation of gases or vapours.** Soc. Anon. des Manufactures des Glaces et Produits Chimiques de Saint-Gobain. Brit. Pat. 747,602; date appl. 17.1.52. France, date appl. 18.1.51 and 19.3.51.—A rectilinear tube, which has its ends closed by transparent walls, is branched off from the conduit through which the gas to be analysed is flowing. One end of the tube is illuminated by an incandescent, u.v. or i.r. lamp (depending on the wavelength of the absorption band of the particular gas to be measured) and a photo-electric cell (sensitive to wavelengths in this band) is located opposite the other end. The current emitted by the cell varies in proportion to the content of gas in the mixture and (after suitable amplification) it may be utilised to estimate the content of this gas in the mixture, at any instant, or to trip a warning relay when this content reaches a predetermined value. The intensity of the light traversing the analysis tube is electrically or mechanically modulated at an audio-frequency, *e.g.*, by use of an a.c. discharge lamp or by use of a rotating sector disc. The moisture content of a gas may be estimated by passing the gas through a column packed with Raschig rings, down which oleum is allowed to trickle, and measuring the opacity of the resulting fog. J. M. JACOBS

2448. **Relationship between interfering substances in flame spectrophotometry.** Shouzow Fukushima, Misako Shigemoto, Isao Kato and Kiyoteru Otozai (Fac. Sci., Univ. Osaka, Japan). *Mikrochim. Acta*, 1957, **1**, 35–70 (in English).—As a first step towards devising an independent flame-photometric method, a systematic and comprehensive survey has been made of the general modes of interference with radiations of Li, Na, K, Rb, Cs, Ca and Ba by more than 20 other constituents (concomitants), including acids, salts and organic substances. The Beckman DU quartz spectrophotometer with 9200 flame attachment and 4030 acetylene - oxygen burner was used. Ambiguous concepts or words are reviewed, and the "specified" $C - I$ curve, the "concomitant" $C - I$ curve, and "additive" and "multiplicative" interferences are defined. A relationship named "rule of log - log translation" has been found to be widely valid. This seems to make feasible some general method of correction for the multiplicative interferences.
D. F. PHILLIPS

2449. **Improvements in spectrographs and monochromators.** Hilger & Watts, Ltd. (Inventor: A. C. G. Menzies). Brit. Pat. 766,909; date appl. 2.7.54.—Light is directed through an entrance slit and on to light-dispersing means serving normally to produce superimposed spectra of different orders, *e.g.*, a diffraction grating or a reflection grating, while between the slit and the grating, or beyond the grating, a second dispersing means, *e.g.*, a prism, is interposed which serves to effect separation of the spectra. J. M. JACOBS

2450. **Semi-automatic arc gap with a motorised stand.** L. E. Owen (Goodyear Atomic Corp., Portsmouth, Ohio, U.S.A.). *Appl. Spectroscopy*, 1956, **10** (4), 203–206.—The continuous manual adjustment of the electrode gap which is necessary in analytical emission spectrography has been obviated by the development of an electrically

controlled, motor-driven, electrode-carrying attachment to the arc-spark stand. The attachment is in the form of arms which are positioned according to signals derived from two adjustable, vertically separated phototransistors located in the image plane of the arc gap produced by a cylindrical lens.
K. A. PROCTOR

2451. **Improved method of introducing samples into the arc flame during work with the activated alternating-current arc.** T. F. Borovik-Romanova (V. I. Vernadskiĭ Inst. of Geochem. and Anal. Chem., Acad. Sci., U.S.S.R., Moscow). *Zhur. Anal. Khim.*, 1956, **11** (5), 634–635.—To avoid sputtering in the arc and formation of spherical agglomerates that do not vaporise, and to ensure complete and uniform evaporation of the sample it is recommended that carbon powder 1·5 to 2 times the wt. of sample (silicate mineral, etc.) be inserted into the crater (4 mm deep and 3·5 mm wide) of a carbon electrode and pressed down with a rod. The sample is then introduced, followed by a small amount of carbon powder.
G. S. SMITH

2452. **The Respectra: A new calculating board for quantitative emission spectrography.** J. W. Anderson (Applied Res. Lab., Glen Rock, New Jersey, U.S.A.). *Appl. Spectroscopy*, 1956, **10** (4), 195–197.—The calculating board can be used for preparing photographic emulsion calibrations and for performing the mathematical steps involved in spectrographic determinations. The transformation of emulsion calibrations into straight lines by the use of Kaiser's modification of the Seidel function is described.
K. A. PROCTOR

2453. **Investigations concerning light sources for spectrum analysis. IX. Electronically controlled high-voltage d.c. spark source with wide frequency range.** A. Bardocz (Central Res. Inst. for Physics, Budapest, Hungary). *Appl. Spectroscopy*, 1956, **10** (4), 183–190.—The apparatus described operates in a frequency range from 10 to 1100 c/s and consists of a spark source and a pulse generator for controlling this source. The spark discharges are controlled by a thyratron tube in parallel with the analytical spark gap, and at the higher frequencies an air jet is applied to the controlling spark gap. The thyratron is operated by the pulse generator, which consists of a free-running multivibrator and a pulse former. The sparking frequencies obtained are independent of the line frequency, but because of its operating principles the spark source is unsuitable for compensating for line voltage variations.
K. A. PROCTOR

2454. **Use of capillary cells [in spectrophotometry].** O. G. Koch (Lab. of Neunkircher Eisenwerk A.-G., Neunkirchen/Saar). *Mikrochim. Acta*, 1957, (1), 30–34 (in German).—The advantages, and in some cases the absolute necessity, of using capillary cells in spectrophotometric micro-determinations, and especially in trace-element determinations, are discussed. The use of such cells is often avoided because of the difficulty of bubble-free filling. A technique is described for a rapid and bubble-free filling which is especially suitable for thick-walled capillary cells (approx. 3 mm wall thickness) with int. diam. 1 to 5 mm and effective cell length of from 5 to 20 cm.
D. F. PHILLIPS

2455. **A temperature-stabilised hydrogen-discharge lamp with high constant intensity.** R. Gerharz (Geo. Washington Univ., Washington, D.C., U.S.A.). *Mikrochim. Acta*, 1957, (1), 125–127 (in German).—The intensity stability of a Nester-type H-discharge lamp (*J. Opt. Soc. Amer.*, 1953, **43**, 432) was studied and improved by a closed-circuit cooling system with thermo-gravitational water circulation. The ultimate fluctuation in intensity was observed to be $< 0{\cdot}03\%$ of the total emission, and this constancy was maintained after more than 600 hr. of intermittent operation.
D. F. PHILLIPS

2456. **An adapter for the Beckman spectrophotometer for the reading of chromatograms and electropherograms.** G. Ceriotti (Centre for the Study and Cure of Tumours, Busto Arsizio, Italy). *Experientia*, 1957, **13** (1), 44–45 (in English).—The cell compartment of the Beckman spectrophotometer is replaced by a box with a large circular hole for entry of the light beam and an adjustable vertical slit in the opposite wall near the photocell. The light beam from the monochromator is collimated into a narrow vertical beam and focused on the slit by means of two cylindrical lenses. A slide mechanism carries the paper strip, which is mounted between two removable glass plates immediately in front of the slit.
A. R. ROGERS

2457. **Applications of infra-red spectroscopy. Application of infra-red continuous analysers.** H. H. Hausdorff (The Perkin - Elmer Corp., Norwalk, Conn., U.S.A.). *Chem. & Ind.*, 1957, (6), 154–158.—Developments in the design and applications of i.r. continuous analysers are described.
K. A. PROCTOR

2458. **Recording infra-red spectra for quantitative analysis.** J. A. Perry, G. H. Bain and W. B. Traver (Standard Oil Co., Whiting, Indiana, U.S.A.). *Appl. Spectroscopy*, 1956, **10** (4), 191–192.—The optimum operating conditions for recording quant. i.r. spectra with the Perkin - Elmer Model 21 spectrophotometer have been determined statistically. Slits should be set as wide as consistent with analytical requirements but short of causing pen oscillation, and zero transmittance should be set with the help of a reference such as a screen. The error is not significantly affected by suppression value and method of slit operation. By using these procedures the spectra can be recorded with a precision of $0{\cdot}25\%$ transmittance.
K. A. PROCTOR

2459. **Bent-crystal X-ray spectrograph.** G. B. Deodhar and R. C. Karnatak (Univ. of Allahabad, India). *J. Sci. Instrum.*, 1957, **34** (1), 21–23.—Details are given of the principle and construction of a new design of transmission-type bent-crystal spectrograph which permits lines to be focused over an extended range by using a curved film holder capable of motion along a line perpendicular to the axis of rotation of the arm carrying the cassette. The crystal is a thin sheet of muscovite mica.
G. SKIRROW

Thermal

2460. **Quantitative analysis with thermal conductivity detection in gas - liquid chromatography.** A. I. M. Keulemans, A. Kwantes and G. W. A. Rijnders (Koninklijke Shell Lab., Amsterdam, Netherlands). *Anal. Chim. Acta*, 1957, **16** (1), 29–39 (in English).—The use of the katharometer as detector in gas - liquid chromatography is discussed, with particular reference to the principles

concerned in its calibration. The practical application of these principles is illustrated with examples in which known mixtures of hydrocarbons are used.
W. C. JOHNSON

Electrical

2461. **Studies on a digital measuring instrument and its application. I. Digital millivoltmeter.** Seishi Takagi and Yoshihiro Maekawa (Osaka Coll. Pharm., Kitayashita, Minamikawachi-gun, Osaka). *Japan Analyst*, 1956, **5** (9), 516–518.—A digital millivoltmeter for < 1500 mV was constructed for use in electrochemical study. The electrical system is similar to the ordinary automatic electronic balancing voltmeter, but the potentiometer drum is connected to a mechanical register with gears, instead of being connected to needles. The accuracy is within $\simeq \pm 0{\cdot}1\%$ and the instrument can be used for potentiometric titration or pH measurement instead of a potentiometer. K. SAITO

2462. **Electrophoresis in a fluid film.** N. Ressler and B. Zak (Wayne County Gen. Hosp., Eloise, Mich., U.S.A.). *Clin. Chim. Acta*, 1956, **1** (4), 392–400.—A description of the method and the results of a study of the variables involved are given. Thermal convection is effectively reduced even at temp. above 32°. Reproducibility is comparable with that of other methods, and mobility is linear with time. Resolution of a number of serum globulin sub-fractions can be attained by electrophoresis for 15 hr. H. F. W. KIRKPATRICK

2463. **Paper electrophoresis in a damp chamber. IV. Continuous electrophoresis and two-dimensional electrochromatography.** Z. Pučar (Inst. "Ruder Bošković," Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1956, **28** (3), 195–209 (in German).—An investigation has been conducted of the movement of charged particles in a continuous paper-electrophoretic process in a damp chamber. Grassmann's method, but with a modified apparatus (described), was used. The movement of an electrically charged particle in two-dimensional electrochromatography comprises the vectorial sum of the horizontal (electrophoretic) and vertical (chromatographic) components. The theoretical aspects are discussed. The term electrochromatography can be applied only to a discontinuous process. The distinctions between continuous electrophoresis, two-dimensional electrochromatography or two-dimensional electrochromatophoresis, and one-dimensional electrochromatography or one-dimensional electrochromatophoresis are indicated. A continuous electrophoretic separation was carried out on a mixture of Tropaeolin O, fluorescein sodium, chlorophenol red, rosolic acid, chrysoidin Y, and nitroaniline, each of these being in ethanolic soln. (50%). The same mixture was submitted to a two-dimensional electrochromatographic separation. The continuous electrophoresis of human serum (undialysed) is also described and illustrated. W. J. WRIGHT

2464. **High-frequency argentimetric titration.** Yu. Yu. Samitov and V. M. Gorokhovskiĭ (V. I. Ul'yanov-Lenin Kazan State Univ.). *Zhur. Anal. Khim.*, 1956, **11** (5), 621–626.—The Q-metric apparatus described earlier (*Anal. Abstr.*, 1957, **4**, 339) is applied to the high-frequency titration of Cl^-, Br^-, I^-, SCN^- and $Fe(CN)_6^{3-}$ in the concn. range $2{\cdot}5 \times 10^{-3}$ to $2{\cdot}5 \times 10^{-5}$ *N*. G. S. SMITH

2465. **A transistor titrimeter.** J. P. Phillips (Univ. Louisville, Ky., U.S.A.). *Chemist Analyst*, 1956, **45** (4), 107–108.—The titrimeter comprises a modified transistor voltmeter circuit with platinum - calomel electrodes, the full-scale deflection on the 50-microamp. meter being made 1 V or less by adjustment of the 10,000-ohm calibration control. The titrator can be made automatic by replacing the meter with a sensitive relay (or better a meter relay), with a solenoid valve as the relay load and fitting the burette into it by rubber tubing. When the current reaches the end-point value, the relay closes and interrupts the titrant flow.
O. M. WHITTON

2466. **A hydrogen - nitrogen gas coulometer.** J. A. Page and J. J. Lingane (The Univ., Toronto, Canada). *Anal. Chim. Acta*, 1957, **16** (2), 175–179 (in English).—A hydrogen - nitrogen gas coulometer (vol. 125 ml) with two pairs of platinum electrodes, 0·1 *M* hydrazine sulphate electrolyte and free from negative error at low c.d. is described. The calibration factor is always const. at within $\simeq 0{\cdot}3\%$ of the theoretical value. A micro-form of this coulometer having a vol. of $\simeq 25$ ml and suitable for the accurate integration of 5 to 20 coulombs of electricity is also described, and its accuracy with different electrolytes is reported.
W. J. BAKER

2467. **Method for indicating the content of a volatile combustible liquid in a mixture containing said liquid, and apparatus therefor.** British Petroleum Co. Ltd., A. R. Javes and C. Liddell. Brit. Pat. 767,127; date appl. 31.3.54.—A mixture of a volatile combustible liquid and an incombustible liquid and/or a liquid which is less volatile than the volatile combustible liquid is brought into contact with a gas (air) stream (*e.g.*, by bubbling the gas through the liquid) which, under the conditions of contacting, does not react with the mixture, at a temp. at which the volatile combustible liquid releases vapour into the gas stream. Thereafter the vapour-laden stream is burnt with a combustion-supporting gas under conditions such that an indication is obtained of the amount of heat evolved during the combustion or of the vol. and/or pressure change accompanying combustion. Preferably, the vapour-laden gas stream is burnt by passage over a platinum-wire combustion catalyst. The platinum wire is made one arm of a Wheatstone bridge circuit and the concn. of the combustible liquid is determined continuously by measuring the change in resistance of the platinum wire (as an indication of the heat involved during the combustion) by means of a galvanometer. The method is particularly suitable for the measurement of alcohols and ketones in industrial waste effluents, *e.g.*, concn. of ethyl methyl ketone in water of the order of 0·05 wt.-% may be satisfactorily detected.
J. M. JACOBS

2468. **Indicator for measuring the carbon monoxide content in a sample of air.** Kollsman Instrument Corp. Brit. Pat. 766,590; date appl. 27.7.54. U.S.A., date appl. 17.9.53.—The indicator comprises a pump for supplying a sample of the air to a reservoir. The reaction system, which is connected to this reservoir, comprises a thermistor - catalyst bank for oxidising the CO and sensing the percentage of CO as a decrease in thermistor resistance, dependent on the temp. rise brought about by the oxidation. The thermistor - catalyst

bank comprises a matched pair of thermistors, each having a nominal resistance of 2000 ohms at 25° and an approx. thermal coefficient of resistance of minus 4% per °C. One of these thermistors is arranged ahead of the catalyst and serves to compensate for small changes in the ambient temp. of the air sample. The pairing of the sensing and compensating thermistors ensures a uniform initial balancing of the Wheatstone bridge measuring circuit in which they are included, with the result that the bridge unbalance is caused entirely by catalytic oxidation of the CO. Temp. changes of $\pm 5°$ produce no significant change in the performance of the indicator. J. M. JACOBS

2469. **The application of the ring-oven technique to radioactive substances.** H. Weisz and F. Scott (Radiochem. Lab., Wolverhampton and Staffordshire Tech. Coll., Wolverhampton, England). *Mikrochim. Acta*, 1956, (12), 1856–1857 (in English).—The extension of the ring-oven technique to the analysis of radioactive substances is described. The sample is concentrated into a narrow ring zone, portions of this zone are then treated with specific precipitating reagents, and washed. When placed in contact with X-ray film, only that portion of the zone which has reacted with the reagents gives an autoradiograph. P. S. STROSS

2470. **A plastics coil simplifying radioactive liquid-phase counting.** S. N. Albert, W. A. Spencer, M. Finkelstein, J. Shibuya, S. Alpert and C. S. Coakley (District Columbia Gen. Hosp., Washington, D.C., U.S.A.). *J. Lab. Clin. Med.*, 1956, **48** (3), 471–475. A two-layered coil is prepared by winding polyethylene tubing (1·77 mm int. diam.) round a glass tube 5 mm in diameter. The coil is placed in a well-type scintillation counter and radioactive fluids are drawn into, or passed through, the coil for counting. The device is used for blood-vol. studies with erythrocytes labelled with ^{51}Cr; when the coil is used as an arterio-venous shunt, continuous blood-vol. estimations may be made. W. H. C. SHAW

2471. **Determination of hydrogen in certain metals by the method of isotopic equilibrium.** A. N. Zaĭdel' and A. A. Petrov. *Zhur. Tekh. Fiz.*, 1955, **25** (14), 2571–2573; *Ref. Zhur., Khim.*, 1956, Abstr. No. 54,782.—Isotopic exchange between the H of the sample of metal and deuterium is obtained in a vessel of vol. V containing D at a pressure p_D. The concn. of H and D in the gas is found spectrographically by means of electrodeless discharge. The content of H in ml in the sample is calculated from the equation—

$$V_0 = (I_H/I_D)_1(p_D/p_0)(T_0/T.V),$$

where $(I_H/I_D)_1$ is the intensity ratio of the lines H_α and D_α, T is the temp. at which p_D is measured, and T_0 and p_0 are s.t.p. The method does not require the use of standards, complete extraction of the H is not necessary, and it has been applied to the determination of H in Zn, Fe and Cr - Ni alloys. G. S. SMITH

2472. **High-sensitivity mass spectrometer for noble gas analysis.** J. H. Reynolds (Dept. of Physics, Univ. California, Berkeley, U.S.A.). *Rev. Sci. Instrum.*, 1956, **27** (11), 928–934.—By using ultra-high vacuum techniques, small samples of rare gases can be closed off within the spectrometer envelope and analysed statically. The detectability of the instrument for Xe is $0{\cdot}5 \times 10^6$ atoms and for ^{36}A it is 14×10^6 atoms. The background spectrum at a spectrometer pressure of 5×10^{-10} mm is described. G. SKIRROW

See also Abstract 2400.

ERRATUM.—April (1957) issue, abstract 1120, line 4. *For* "J. Vavruch" *read* "I. Vavruch."

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	%(v/v)
per cent. (wt. in vol.)	%(w/v)
per cent. (wt. in wt.)	%(w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_B
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{3+}, Fe^{2+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

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